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PART II

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THE X-RAY EXAMINATION OF MATERIALS IN INDUSTRY

EDGAR MARBURG LECTURE

BY GEORGE L. CLARK[1]

It is with a mingled feeling of profound humility and of deep gratification that we come to honor the memory of a great engineer. It is not that his work and his personality are forgotten by the Society except on a day in June each year, nor is this memorial a funeral oration, nor a saddened recapitulation of the past, for such a tribute would not be in harmony with the living spirit of the man which envisioned the future and its glorious progress towards perfection in all things. No man could be more deeply interested in the application of new scientific tools to industry and to the establishment of new standards than was Edgar Marburg, as evidenced by his faithful service for the American Society for Testing Materials. Thus, no more appropriate or eulogistic subject for the Edgar Marburg Memorial Lecture in 1927 could have been selected than the newest industrial tool, X-rays, discovered only three decades ago, and until now known only in hospitals or as a curiosity in academic laboratories of physics. It is distinctly the greatest honor and privilege which may come to any man to be called upon to deliver as this memorial lecture an exposition of the achievements and the signs of promise of a new and fundamentally scientific method of examining materials. By the linkage of the memory of Marburg with the unquenchable search for truth do we honor both the man and the search.

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[1] Associate Professor of Chemistry, University of Illinois; formerly Assistant Professor of Applied Chemical Research and Divisional Director of the Research Laboratory of Applied Chemistry, Massachusetts Institute of Technology.

X-RAYS

X-rays are electromagnetic vibrations propagated as waves or as bundles of radiant energy through space without the passage of matter. In every respect are they identical with ordinary light except that the wave-lengths are only $\frac{1}{10{,}000}$ as great, on the average, as visible light. In the same way, the extremely penetrating cosmic rays which come from the far reaches of the universe, with wave-lengths only $\frac{6}{100{,}000{,}000}$ as long as yellow light, the γ-rays of radium, the ultra-violet rays, infra-red heat rays, and radio waves are all like light except in wave-length. In all this great range of the electromagnetic spectrum from waves only 0.0004×10^{-8} cm. up to those hundreds of meters long, the human eye is sensitive only to that extremely narrow band which we call light. Because X-rays are shorter than light, it follows that they are more penetrating, that they should pass through materials opaque to light, and that they

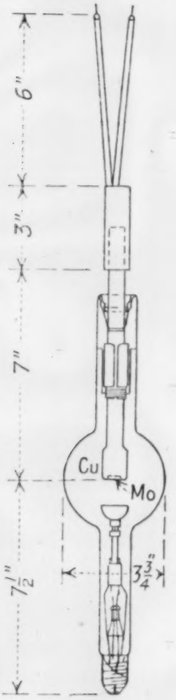

FIG. 1.—Diagram of Coolidge-type X-ray Tube.

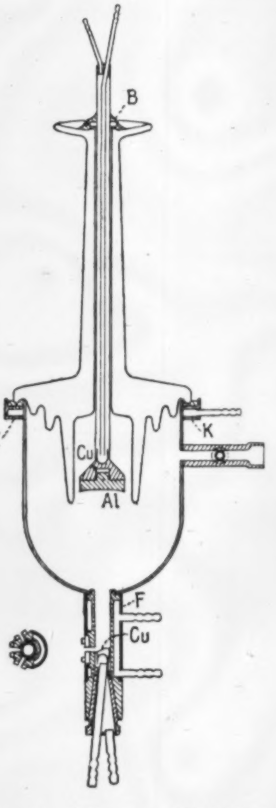

FIG. 2.—Diagram of Hadding-type X-ray Tube.

should be associated with a finer subdivision of matter than is apparent by examination in visible radiation. As a matter of fact, we find that X-ray wave-lengths are of the same order of magnitude as the size of the ultimate atoms of matter. By means of X-radiation, the messenger, therefore, the knowledge of the true unit building blocks of matter is gained.

X-rays are produced whenever a stream of negative electrons, in an evacuated bulb under the acceleration of a high voltage, bombard matter. As these unit particles of negative electricity are suddenly stopped, their energy of motion is converted into radiation energy and X-rays are emitted from the bombarded target. In the Coolidge-type tube (Fig. 1) the source of electrons is a hot-wire filament; in other tubes widely used in industrial applications such as the Hadding-Siegbahn tube (Fig. 2) residual gas is left in the tube and under the electric discharge the molecules are ionized, bombard the cathode and liberate electrons, which in turn bombard the anode or target. A beam of X-rays may be analyzed into a spectrum by a suitable grating such as any crystal, just as a prism or a grating of finely ruled lines on glass will spread white light into a spectrum of component colors. Two types of X-ray emission spectra are recognized. One is a continuous band with many wave-lengths with a short wave-length limit which depends only upon the voltage on the tube and not upon the material of the target; the other is comprised of series of sharp lines whose wave-lengths are distinctly characteristic of the material of the target in the tube. Thus, from a tungsten-target tube the most intense characteristic ray has a wave-length of 0.20885 Å unit;[1] molybdenum 0.70780; copper 1.53726, etc. As a matter of fact, if the square roots of the reciprocals of the wave-lengths of this strongest line (the $K\alpha_1$) or of other corresponding lines, should be plotted against the atomic numbers of elements from hydrogen (1) to uranium (92), a nearly straight line is obtained. This fundamental relationship called the Moseley Law is the basis for much of our knowledge concerning the structure of atoms, and by means of it also unknown elements are discovered. In discovering element 61, illinium, Hopkins at the University of Illinois, interpolated on the straight lines for the theoretical characteristic X-ray wave-lengths and then fractionated his materials until these spectral lines appeared. It is at once evident that a very exact method of analysis of unknown substances is afforded by making them the targets of X-ray tubes and measuring the wave-lengths of the characteristic X-rays which they emit. Similarly, the characteristic absorption of X-rays may identify the element in specimens used as absorbing screens placed in a beam. For the examination of materials, however, it is most convenient to use X-rays of known wave-lengths from a permanently constructed X-ray tube. In this way it is possible to examine all materials for gross structure and the presence of homogeneities or defects—the so-called science of radiography—and also for fine structure even down to the positions of atoms in space.

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[1] One Ångström unit is equal to 10^{-8} cm.

FIG. 3.—Radiographic Examination of Cast Steel Showing Shrinkage Pipe. (Courtesy Dr. H. H. Lester.)

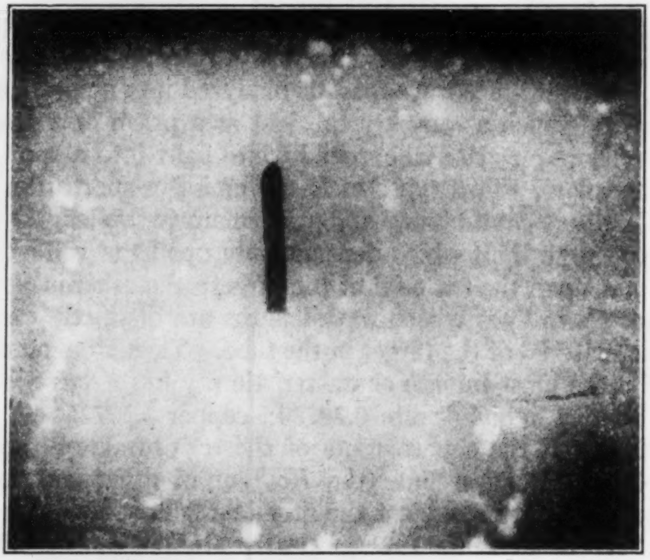


FIG. 4.—Radiograph of Cast Steel Showing Imperfections. (Courtesy Dr. H. H. Lester.)

RADIOGRAPHY

While X-rays penetrate matter opaque to light, they are absorbed in accordance with a simple exponential law. Any inhomogeneity or defect in a material will have a different density and absorbing power for X-rays than the main body of material. Hence, the X-rays which pass through a material will have varying intensity and when they strike a photographic plate, a "Shadowgraph" of the specimen is registered, which may also be visually observed on a fluorescent screen. Observation of bones in the body and teeth is, of course, the

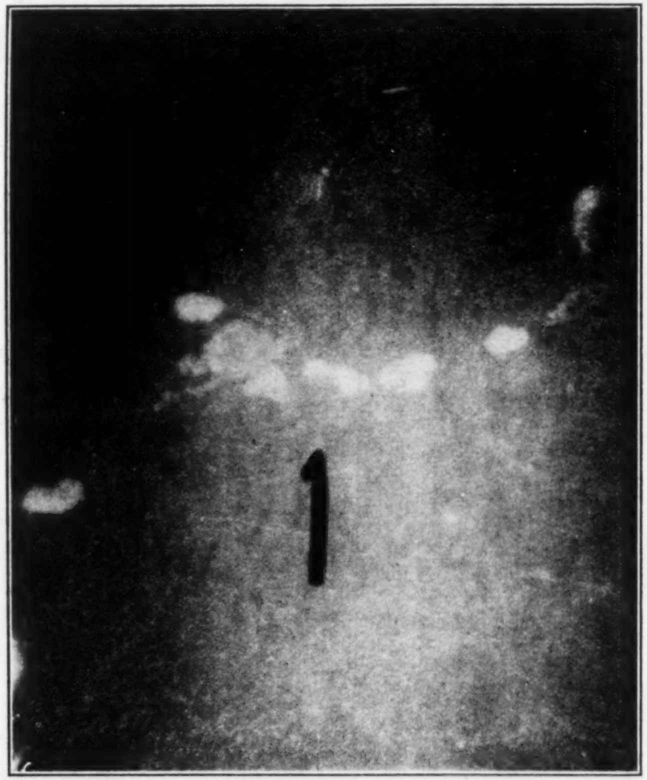


FIG. 5.—Radiograph of Cast Steel Showing Imperfections. (Courtesy Dr. H. H. Lester.)

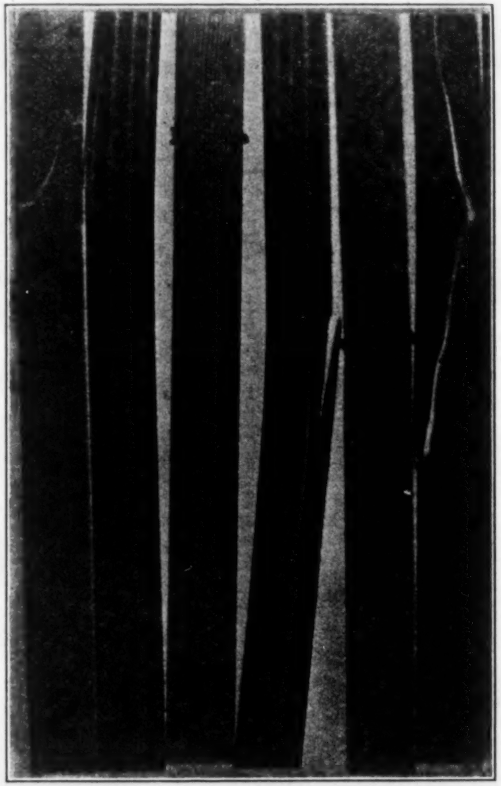


FIG. 6.—Radiograph of Ash Lumber Used in Airplane Wings. (Courtesy Dr. H. H. Lester.)

most familiar example of radiography. The experimental technique is comparatively simple since it consists merely in placing an object to be radiographed between an X-ray tube and a photographic plate. There are many details, however, which require long experience. The higher the voltage on the X-ray tube, the shorter is the effective wave-length and the more penetrating the beam. The maximum thickness of steel which may be radiographically examined is 4 in. Further advances can come only with newly designed X-ray tubes capable of withstanding higher than 300,000 volts. There are many important, interesting and useful applications of this branch of X-ray science. The following are some which have been found in America.

Radiographic Applications:

1. Medical and dental diagnosis, identifications of persons, shoe fitting.
2. Metal castings for internal gas cavities, sand and slag inclusions, pipe cavities, porosity, cracks, metal segregation.
3. Welds, for soundness.
4. Coal, control of washing process.
5. Minerals, classification.
6. Rubber, symmetry of golf-ball centers, adhesion to cords in tires, inspection of reclaimed rubber for nails, heavy metal content.
7. Insulated wire inspection.
8. Grenades and shells, filling.
9. Wood used in airplanes, etc., for cracks, wormholes, knots.
10. Hidden wires and pipes in walls, mystery packages, contraband.
11. Internal diameters of metal pipes and capillaries, height of capillary rise of liquids, clogged gasoline feed lines.
12. Position of electrodes in vacuum tubes.
13. Old paintings, examination for retouching and superposed pictures.
14. Glass, heavy metal content.
15. Pigments, partial identification.
16. Swiss cheese, size and location of holes.

Particular attention is called to the remarkable developments in casting and welding technique which have been made possible by radiography. The performance of equipment subjected to high temperatures and high pressures such as in oil stills and power plants is thus assured. In Figs. 3, 4 and 5, typical radiographs of internally defective steel castings are reproduced, and in Fig. 6 this method of examining ash lumber for use in airplane wings shows clearly the distinction between brash and good wood.

THE FINE STRUCTURE OF MATERIALS

I. X-RAYS AND CRYSTALS

The examination of materials for ultimate structure and constitution depends upon the fact that crystals are three-dimensional diffraction gratings for X-rays. Crystals are built up of parallel planes upon which lie the atoms, and these planes are spaced from each other at a distance which is entirely comparable with the wavelength of X-rays. Thus, a structure which appears homogeneous to the eye will disclose a fine structure in the microscope, which in turn will show subdivision in the ultra-violet microscope, and one of these subdivisions will diffract X-rays in a way which indicates that it in turn is built of ultimate crystal units. Thus, when a beam of X-rays defined by small pinholes passes through a single crystal grain of iron, the result is a pattern of sharp spots (Fig. 7) on a photographic plate placed behind the crystal. In every direction through the grain are parallel planes of atoms with a certain spacing. Each set of planes picks out from the X-ray beam impinging upon this set at a certain angle a ray of a definite wave-length, λ, which is reflected in accordance with the simple Bragg law $n\ \lambda = 2\ \delta \sin \theta$. This diffraction

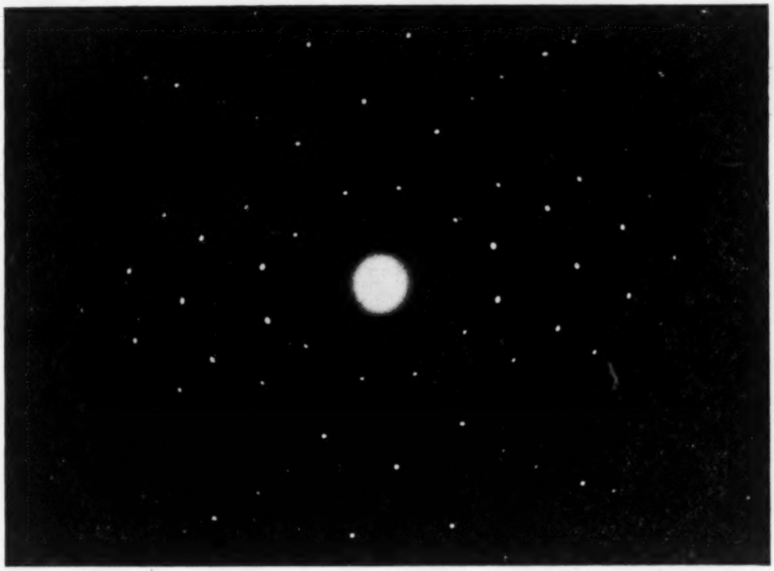


FIG. 7.—Typical Pattern for a Single Crystal of Iron (Beam Parallel to a [110] Direction).

pattern upon analysis proves that the smallest subdivision which is still crystallin iron is a cube with an atom of iron at each corner and one at the center and with an edge length of 2.86×10^{-8} cm. (Fig. 8). This minute unit cell multiplied in all directions produces the visible crystal grain. With such a fundamental relationship between matter and X-radiation it is evident that crystals of known unit cell dimensions may be used as gratings to analyze beams of X-rays, and, *vice versa,* X-rays of known wave-lengths may be used to analyze the unknown structure of crystals. Even in liquids, colloidal gels and other so-called amorphous substances without vestige of crystal faces or of geometric arrangement in space, the fact remains that molecules or aggregates of molecules in disordered motion cannot approach each other nearer than a certain limit. This distance of closest approach may be ascertained from the simple X-ray diffraction patterns of one or two broad rings. X-rays are a versatile tool and the information which they vouchsafe concerning fine structures of materials is astonishingly complete, as shown by the following enumeration:

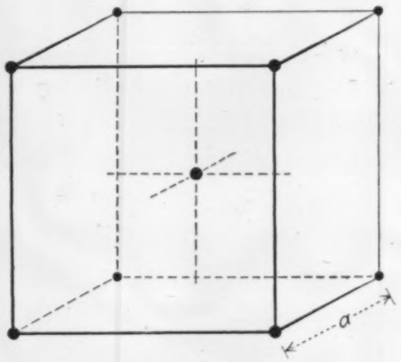


FIG. 8.—Ultimate Crystal-Unit of Iron.

The ultimate crystal of iron is a cube with one atom at each corner and one atom at the center. This minute unit-cell is multiplied in all directions to form the visible crystal.

Types of Diffraction Information:

1. Crystallin or amorphous.
2. Macrocrystallin or colloidal.
3. Lattice structure, atomic and molecular arrangement and dimensions for crystals, distances of nearest approach of molecules or aggregates in liquids, gels, etc.
4. Chemical identity (compound, mixture, adsorption complex, solid solution), purity, phase structure.

5. Transformations and allotropic modifications.

6. Chemical change, aging.

7. Single crystal or aggregate.

8. Random or fibered aggregate.

9. Degree of fibering deformation and quantitative analysis of mechanism of deformation; effects of fabrication—rupture, rolling, drawing, torsion, impact, complex deforming forces, electrodeposition, annealing, directional properties.

10. Grain size.

11. Internal strain.

12. Differentiation between surface and interior structure.

13. Thickness of films.

14. Effect of low and high temperatures and high pressures, and the nature of recrystallization.

15. Uniformity of structure in different lots, in the same lot, in the same unit.

16. Differentiation fundamentally between same materials, with satisfactory and unsatisfactory properties for a given practical purpose.

II. THE PINHOLE DIFFRACTION METHOD

1. *Attributes of a Desirable Method:*

Certain attributes are largely desirable in any diffraction method which may serve as a research and testing tool for the foregoing purposes. These are:

(*a*) Simplicity and relatively low expense; absence of moving or complicated apparatus requiring expert control and measurement; time saving; and possibility of simultaneous multiple exposures with one X-ray tube.

(*b*) Elasticity to meet varied conditions and types of specimens.

(*c*) Minimum preparation of specimens so that they may be examined in their exact condition without variations introduced by polishing, powdering, placing in containers, etc.

(*d*) Single diffraction patterns as nearly complete as possible (that is, 360 deg. in azimuth around the direct X-ray beam zero position), so that more than one crystallographic zone may register simultaneously and successive resettings of specimen may be eliminated.

2. *Description of Method:*

Among the dozen or more possible experimental arrangements, the single method which most satisfactorily yields information of the 15 types enumerated above and which most successfully combines all the desirable attributes of an experimental tool is the pinhole method.

In Fig. 9 is illustrated the essential experimental arrangement. The X-ray beam, defined by 2 pinholes in lead blocks or other suitable material, passes through a specimen placed over the outer pinhole, or is reflected from the surface of the specimen (Fig. 10); the diffraction pattern is recorded on a photographic plate or film usually flat and perpendicular to the beam at a fixed distance from the specimen.

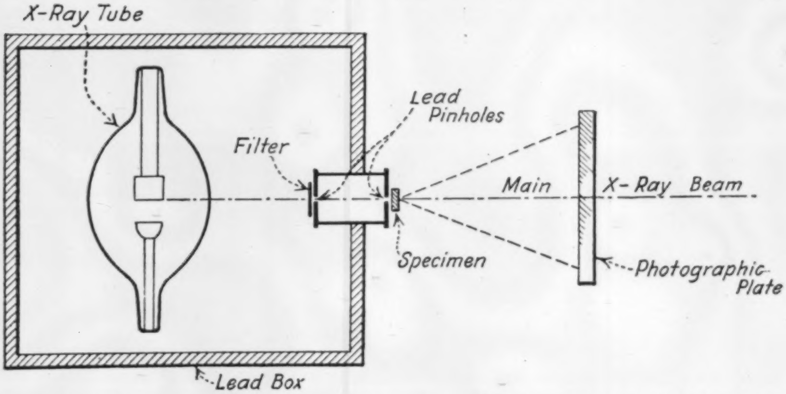

X-Ray Tube
Lead Pinholes
Filter
Main X-Ray Beam
Specimen
Photographic Plate
Lead Box

FIG. 9.—Diagrammatic Sketch of Apparatus Used to Obtain Pinhole X-ray Diffraction Patterns.

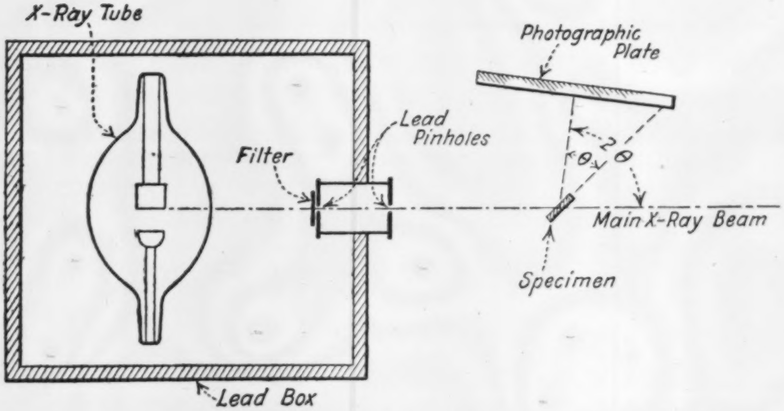

X-Ray Tube
Photographic Plate
Lead Pinholes
Filter
2θ
θ
Main X-Ray Beam
Specimen
Lead Box

FIG. 10.—Diagrammatic Sketch of Apparatus Used to Obtain X-ray Surface Reflection Patterns.

The method is one of extraordinary simplicity and flexibility. When the beam from any X-ray tube is used unfiltered there are many wave-lengths; the pinhole method employing a polychromatic beam is simply the classical Laue method, useful primarily with single crystals as demonstrated for the single grain of iron in Fig. 7. When, however, the beam of X-rays is filtered (for example, by means of a screen of zirconium oxide for molybdenum rays) so as to absorb all but a sensibly monochromatic ray, the method may be termed *monochromatic* pinhole. It yields simpler patterns for single crystals and is applicable to powders, aggregates, fibers and amorphous substances. There are many modifications: surface reflection (Fig. 10); back reflection from very large specimens; cylindrical films co-axial with the specimen at the center, or co-axial with the beam; rotating specimen or pinhole, etc.

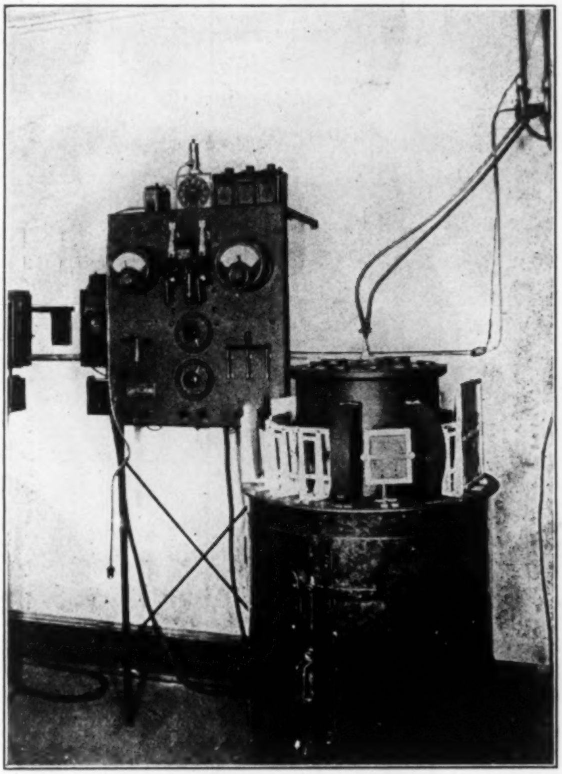
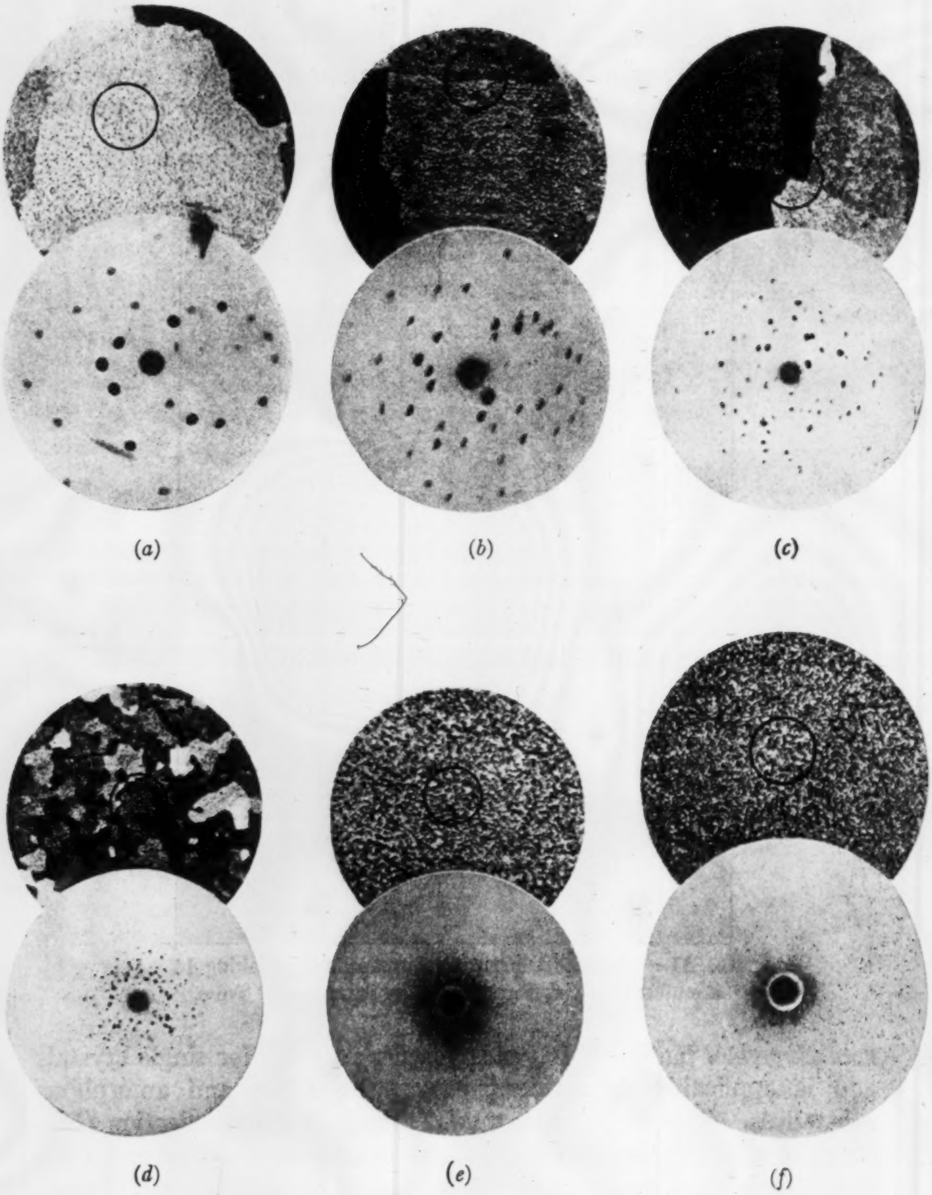


FIG. 11.—Multiple Diffraction Apparatus for Making 18 Simultaneous Exposures from the Same X-ray Tube.

For economy of time and expense in research and routine examinations of commercial metals it is obviously of great value to be able



(a) (b) (c)

(d) (e) (f)

FIG. 12.—Patterns for Simultaneous Diffraction of X-rays by Aluminum.

to make several exposures simultaneously with one X-ray tube. The first multiple diffraction apparatus combining the various modifications of the pinhole method and the Hull method is shown in Fig. 11. The apparatus is fully described in a recent paper;[1] commercial equipment of similar design is now on the market. The apparatus is versatile in that several diffraction methods may be employed simultaneously and in that pinholes and slits are made removable, or adjustable to the target of the X-ray tube with its long axis vertical. and interchangeable. Such an apparatus may be operated continuously day and night and thus make possible accumulations of a mass of data in a comparatively short time.

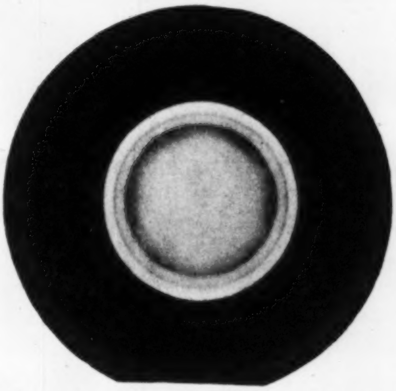


FIG. 13.—Pinhole Diagram of Small-Grained Random Aggregate of α-Iron.

3. Typical Patterns:

(*a*) *Single crystal.*—Fig. 7.

(*b*) *Superposed grains, and aggregates; effects of grain size.*—A metal specimen which consists of one crystal grain is distinguished by a regular pinhole pattern of diffraction spots, showing a symmetry of number and arrangement on zone ellipses which cannot be obscured, even though the X-ray beam does not pass through the grain parallel to a principal axis. If the X-ray beam passes through two grains, the patterns on the plate are superposed; as the number of grains lying side by side or under one another in the path of the beam increases, the spots become more numerous and smaller, all traces of the zone ellipses for one grain being obscured. At the same time the intensity decreases, and longer times of exposure are required. A condition of minute size is finally reached where on a Laue diagram there appears an almost continuous darkened ring, and in the monochromatic pinhole method the concentric rings of uniform intensity. Figure 12 gives the patterns for the simultaneous diffraction of X-rays by 1, 2, 5, 120, 2000, and 1,000,000 grains of aluminum in the path of the beam. Figure 13 shows the typical concentric ring patterns for iron with a particle size between 10^{-3} and 10^{-6} cm. (for this specimen the size is at the lower limit). This pattern is a special modification of the straight line spectra obtained by the familiar Hull-powder method in which the monochromatic beam is defined by slits instead of pinholes (Fig. 14). In general, a metal aggregate with particle size larger than 10^{-3} cm. produces dashed or spotted patterns with the individual maxima lying on rings for the lower size limit (Fig. 15). The range of particle sizes between 10^{-3} and 10^{-6} cm. cannot be differentiated ordinarily from diffraction patterns since the rings or lines are uniformly sharp and continuous. For sizes smaller than 10^{-6} cm. the widths of the diffraction interferences increase until a given amount of energy is spread so widely that maxima may no longer be identified. Particle size may be calculated from the equation, deduced by Debye and Scherrer and found to be experimentally satisfactory:

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[1] Clark, Brugmann and Aborn, *Journal*, Optical Soc. Am., Vol. 12, p. 379 (1926).

FIG. 14.—Diffraction Spectrum of α-Iron by Hull Method.

$$B = 2\sqrt{\frac{\log_e 2}{\pi}} \cdot \frac{\lambda}{D} \cdot \frac{1}{\cos \theta/2} + b$$

where B is the angular breadth of a diffraction halo measured between points of half-maxima intensity, θ the diffraction angle, λ the wavelength, D the average thickness of the crystal parallel to a cubic axis, and b the minimum breadth determined by the particular apparatus. This equation has been used in the evaluation of particle size in martensite, in colloidal metal preparations, and in metallic catalysts as related to activity. It has been applied even to localized intensity maxima on fiber diagrams to determine the size of colloidal micelles (for example, rubber, 90 Å or 4000 $(C_5H_8)_8$ groups). As an extreme case of fragmentation is shown the structure of the outermost layer of a cold-rolled aluminum sheet taken by the surface reflection method (Fig. 16).

(*c*) *Fiber Structure.*—There is an intermediate state between the single crystal and a chaotic aggregate of small crystals: namely, one in which the small crystals are oriented in a definite fashion with respect to a common direction.

All natural fibers (asbestos, cellulose of all varieties, silk, etc.) are constructed in this way so that they yield characteristic fiber diagrams. Figure 17 shows a typical pattern for chrysotile asbestos taken with the beam perpendicular to the fiber axis. That this is a a perfect fiber is shown by the fact that the localized intensity maxima lie on hyperbolas, as well as on the less distinct Hull-Debye-Scherrer rings. As fibering orientation is less perfect, the rings become more prominent than the hyperbolas, the spots become arcs of these rings, and in the limiting case of no orientation the usual continuous rings appear. "Fibering" in various degrees can be artificially produced, most notably in metals, by mechanically working a chaotic aggregate, that is, rolling into a sheet, drawing into a wire, twisting, compressing, etc. Figure 18 reproduces the fiber diagram of a cold-drawn aluminum wire; Figure 19 of a cast steel bar ruptured in a tension testing machine. Studies of this kind have been made of temperatures from −100 to +1600° F. An interesting feature is the clear delineation of the "blue-brittle" range in the X-ray patterns.

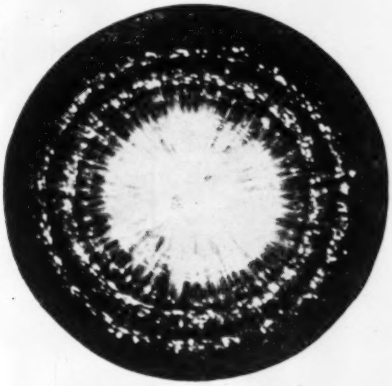


FIG. 15.—Pinhole Diagram of Coarse-Grained Random Aggregate.

It does not follow that orientation of the grains takes place in all directions. Figure 20(*a*) represents the structure when the X-ray beam is perpendicular to the rolling direction in a copper sheet, while Fig. 20(*b*) was taken with the beam parallel to the rolling direction. The first shows fibering (imperfect) while the second shows random arrangement. These distinctly directional properties are further illustrated in Figs. 21(*a*) and (*b*) for surface reflection patterns from rolled duralumin sheet; for the first the beam impinged on the surface in the direction of rolling; in the second the beam struck the same spot on the same specimen turned through 90 deg. in the plane of the sheet.

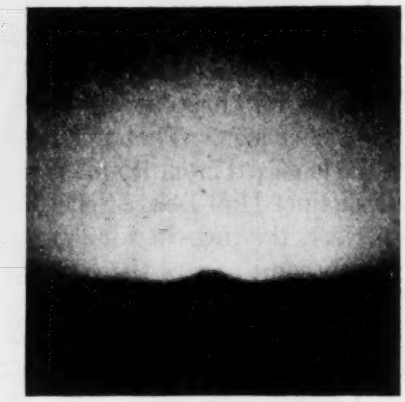


FIG. 16.—Surface Reflection Pattern for Cold-Rolled Duralumin Sheet with the Beam Barely Grazing the Surface, Illustrating Extreme Fragmentation of Surface Layer During Working.

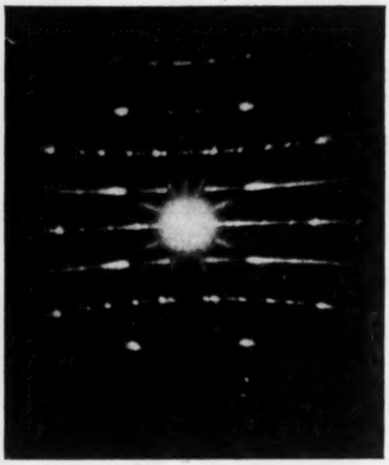


FIG. 17.—Typical Monochromatic Pinhole Pattern for Perfect Fiber (Asbestos), as Shown by Hyperbolas.

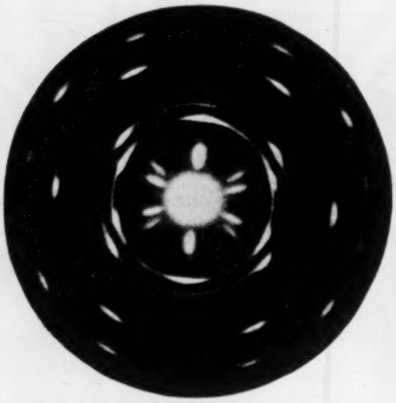


FIG. 18.—Pattern for Cold-Drawn Aluminum Wire, Showing High Degree of Fibering.

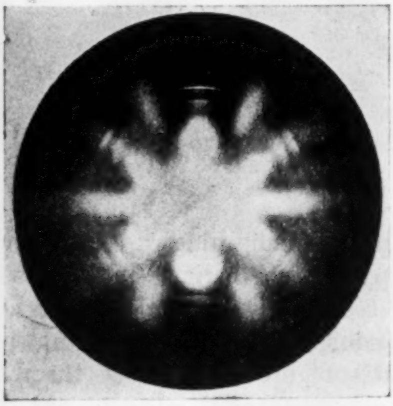
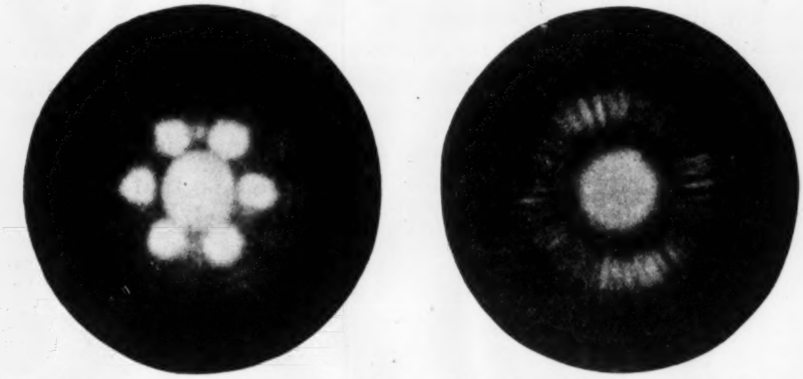


FIG. 19.—Fiber Pattern for Cast-Steel Bar at Point of Rupture in a Tension-Testing Machine.

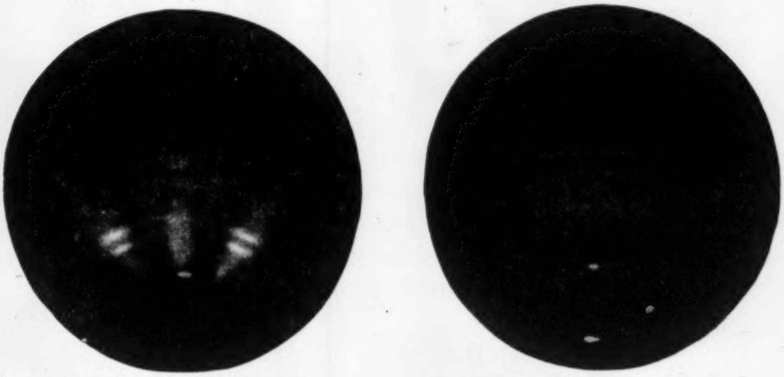
The study of mechanical deformation in metals represents, therefore, one of the great achievements of the pinhole method. The entire diagram 360 deg. in azimuth is obviously highly desirable and consequently the method is superior in ascertaining this type of information to any other. As will be illustrated in specific cases, the behavior of any metal, even after heat treatment, is dependent on the mechanical work which induces fibering and directional properties. In many of the studies of commercial metals, unsatisfactory behavior is to be attributed to faulty mechanical working and to faulty annealing designed to remove directional properties.



(*a*) Taken with Beam Perpendicular to Direction of Rolling.

(*b*) Taken with Beam Parallel to Direction of Rolling.

FIG. 20.—Patterns Illustrating Directional Properties for Same Specimen of Rolled Copper.



(*a*) In Direction of Rolling.

(*b*) With Specimen Turned Through 90 deg.

FIG. 21.—Surface Reflection Patterns for Rolled Duralumin Sheet with Beam Reflected from Same Spot on the Specimen.

Aside from these practical aspects of deformation, there are several important fundamental points of interest. A fiber diagram is actually more easily interpreted than ordinary continuous ring patterns. One fundamental crystal spacing or identity period in the direction of fibering deformation may be calculated very simply from the hyperbolic loci, entirely independently of any knowledge concerning crystallographic system, diffracting plane indices, etc. It is advantageous in some cases to attempt to fiber a material on this account.

Furthermore, by the use of one of several proposed methods[1] the fiber diagrams may be quantitatively interpreted so that the actual mechanism of deformation of metals may be deduced.

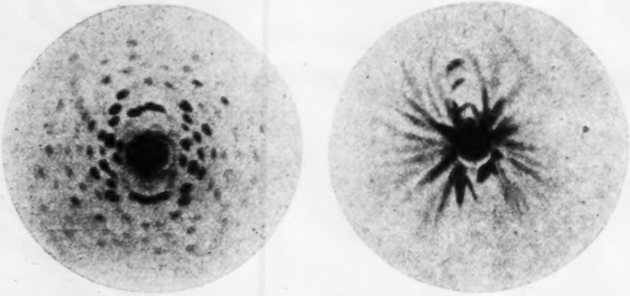


FIG. 22.—Effect of Bending Distortion upon Laue Pinhole Pattern of a Single Gypsum Crystal (Czochralski).

In the simplest and most usual case the angle, δ, measured upon the film between a vertical zero line (or reference line parallel to the fiber axis in a wire) and a line joining an intensity maximum to the center is also the angle α between the normal to a set of diffracting planes in the wire and the fiber axis. Each Debye-Scherrer ring, corresponding to the [111], [100], etc., planes will have its own maximum at characteristic values of δ. From such considerations it is possible to determine the crystal planes which are parallel and perpendicular to the direction of drawing or rolling, which lie in the surface of a sheet, etc.

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[1] Polanyi and associates, *Zeitschrift Physik*, Vol. 7, pp. 181, 197 (1921); Vol. 9, p. 123 (1921); Vol. 10, p. 41 (1921).

Uspenski and Konobejewski, *Zeitschrift Physik*, Vol. 16, p. 215 (1923).

Glocker and Kaupp, *Zeitschrift Physik*, Vol. 24, p. 121 (1924).

Wever, *Zeitschrift Physik*, Vol. 29, p. 69 (1925).

Bozorth, *Physical Review*, Vol. 26, p. 390 (1925).

Konobejewski, *Zeitschrift Physik*, Vol. 39, p. 415 (1926).

Glocker, *Zeitschrift Physik*, Vol. 31, p. 386 (1925).

This information has an important bearing upon practical properties of metals in terms of ultimate crystal units and even atoms—hardness, elastic constants, tensile strength, recrystallization mechanism, etc.; consequently, it is fundamentally related to the scientific control of heat treatment of metals. In the next section some of the important results already deduced from such patterns will be reviewed.

(*d*) *Internal Strain.*—In an uninjured crystal lattice, the planes are in fact plane and act like very good little mirrors towards X-rays, so that images of the narrow aperture produced by the planes are sharp elliptical spots on the photographic plate. If, however, the crystal is stressed beyond the elastic limit, either through external forces or through thermal stresses, translation on slip planes occurs, which as a rule is accompanied by simultaneous bending around crystallographic directions. As a result of this, the planes are curved approximately in the shape of a cylinder, so that the reflection images become distorted into streaks or asterism striations as they do in a cylindrical mirror.

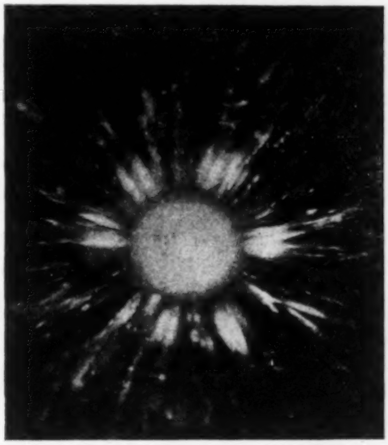

FIG. 23.—Irregular Radial Asterism Striations Produced by Internal Strain in a Metal Aggregate (Steel Casting).

Conversely, it may be concluded from the occurrence of such asterism striations that the space lattice has been stressed beyond the elastic limit and that there are residual inner elastic stresses as a result of translation and bending in the lattice, which can be equalized only by suitable heat treatment.

This phenomenon was first demonstrated by X-rays in the study of mica by Rinne. Mica may be bent purely elastically, whereupon the Laue spots, typical of the unbent crystal, change their appearance markedly by losing their sharpness and by elongating radially. Czochralski[1] showed the same type of phenomenon in plastically deformed single crystals of aluminum and gypsum. In Fig. 22 are reproduced the Laue photographs of a normal and a plastically bent gypsum crystal, respectively, showing clearly the asterism. The same type of change occurs when single metal crystals are stretched, compressed, bent, or subjected to torsion or to any combination of these deformations. Usually the resultant diagram shows little or no symmetry of the radial streaks, since different planes (and their interference spots) are differently affected.

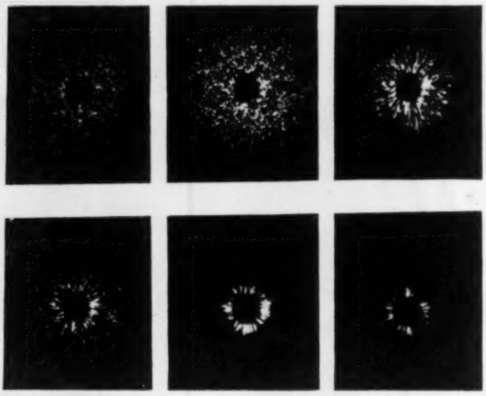

FIG. 24.—Effects upon Pattern of Successive Loading of Specimen of Silicon Steel, Illustrating Asterism Striations as Evidence of Internal Strain.

Not only do asterism striations indicate distortion in single crystals but they also appear for aggregates in a state of strain, particularly prominently when the X-ray beam contains some general radiation with wave-lengths shorter than that associated with sharp ring interferences. Cast metals which are severly quenched show these streaks even when there is absence of preferred orientation of grains. Figure 23 is an illustration for the case of cast steel. Furthermore, it is possible to apply loading stress upon a specimen showing no streaks and make these appear. Figure 24 presents a remarkable new series of photographs showing the effect of successive loading of a specimen of silicon steel. The pattern is extremely sensitive for such a condition—far more so than any other method of detection of internal strain. For this reason it is practically impossible to determine numerical values from an asterism diagram. However, by proper heat treatment it is possible to remove practically all traces of this condition; consequently, the information is of immeasurable value from a practical point of view.

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[1] *Zeitschrift Metallkunde*, Vol. 15, p. 60 (1923).

The question is still unanswered as to just how great internal distortion can be endured by a specimen and still prove practically satisfactory. Controlled experiments now under way in which asterism striation diagrams are related to changes in outer dimensions of a metal specimen from which successive inner layers are removed (or, *vice versa*, changes in inner dimensions with removal of outer layers), should go far towards standardizing the important problem of internal strain. The great difficulty in the quantitative interpretation of X-ray patterns lies in the necessity of separating the effects of grain size, preferred orientation of grains introduced by working, and true strain or distortion such as is introduced by rapid quenching, by separation of new phases in an alloy during aging, or by working so severe as to transcend elastic deformation. Much research work must be done along these lines, particularly as they concern the causes of hardness.

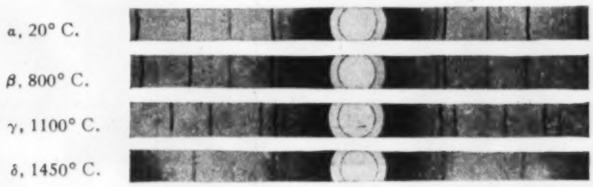

FIG. 25.—Patterns for the Allotropic Modifications of Iron.

III. SOME FUNDAMENTAL RESULTS IN THE SCIENCE OF METALS OBTAINED BY THE PINHOLE DIFFRACTION METHOD

1. The Crystallin Constitution of Metals and Alloys:

While lattice dimensions and constitution may be derived from simple pinhole diffraction patterns for fine-grain and aggregates these data can be determined with probably greater accuracy by other methods, by the Hull method, for example, for here the crystal spectrum lines are straight, sharp, more widely separated, and their displacements easily measured. For single crystals the Laue and monochromatic pinhole methods are still frequently used, but for more complicated structures in alloy systems the rotating crystal method, yielding a complete diagram, is now generally preferred, particularly since methods of quantitative interpretation have been published by Bernal.[1]

Since information on lattice structures is easily available,[2] only the briefest recapitulation will be given here:

1. Pure metals: Report for 1926 of Sub-Committee VI, on X-ray Metallography, of Committee E-4 on Metallography, *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part I, p. 444 (1925).

2. Allotropic modifications: Iron (Fig. 25), cerium, cobalt, tin, manganese, thallium, chromium. Particular interest is centered in manganese, recently studied by Westgren and Phragmen[3] in that 56 atoms are assigned by them to the unit cube of α-Mn and 20 for β-Mn.

3. Intermetallic compounds: About two dozen have been more or less uniquely analyzed.

FIG. 26.—Crystal Spectra for Pure Copper (Above) and 80–20 Brass (Below) Illustrating Solid Solution and the Expansion of the Copper Lattice by Substitution of Zinc Atoms.

4. Alloys formed by a continuous series of solid solutions: A dozen binary systems for pairs of metals crystallizing on the same lattice, illustrate the approximate Vegard additive law that lattice parameters are linearly related between the limits of the pure metals for percentage composition in atomic percent.

5. Alloys forming discontinuous solid solutions, but no compounds: For example, iron-nickel, lead-tin, nickel-chromium.

6. Alloys forming no solid solutions: Magnesium-tin and magnesium-lead.

7. Non-ferrous alloys involving solid solutions and compounds—notably the complete systems copper-aluminum, and copper-zinc (brass), studied both by Owen and Preston[4] and Jette, Westgren and Phragmen.[5] For the latter system there is essential agreement

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[1] *Proceedings*, Royal Society, 113A, 117 (1926).

[2] International Critical Tables, Vol. I; Clark "Applied X-rays," Chapter XX.

[3] *Zeitschrift Physik*, Vol. 33, p. 777 (1925).

[4] *Proceedings*, Physical Society (London), Vol. 36, pp. 14, 49 (1924).

[5] *Journal*, Inst. of Metals, Vol. 31, p. 193 (1924); *Philosophical Magazine* (VII) 1, 311 (1925).

except for the γ-phase. Owen and Preston assign a rhombohedral structure, and Jette, Westgren and Phragmen a cubic unit containing 52 atoms. This extraordinary result has had surprising support from the work of Bradley and Thewlis[1] who show that if 2 atoms are removed from a simple combination of 3 concentric body-centered cubes, the structure of γ-brass results. Figure 26 shows the comparison of the spectra of pure copper and of a solid solution of 80 per cent copper and 20 per cent zinc. The patterns are identical except that the lines for the alloy are shifted, indicating an expansion of the unit crystal cubes.

8. Iron-carbon alloys: Austenite (γ-iron lattice), cementite (orthorhombic), and martensite (α-iron lattice with diffuse lines) have been studied although agreement as to the interpretation and significance of the results is not yet attained, particularly in the case of martensite.

Fink and Campbell[2] in their studies of drastically quenched eutectoid and hypereutectoid steels found evidence of a body-centered tetragonal structure which Bain considered to be an arrested stage in the allotropic change from γ- to α-iron. This important discovery has received very recent complete confirmation in the work of Selyakov in Russia.[3]

9. Complex Alloys: Bain[4] from X-ray studies of hardenable copper alloys containing silicon, nickel, cobalt and chromium was able to show that hardening of the alloy is due to slight lattice distortion produced by the absorption of alloying atoms, sufficient to oppose smooth slip on atomic planes. Bain and Griffiths[5] identified a new and complex constituent in alloys of iron, chromium and nickel at temperatures between 800 and 950° F., and solid solutions above 1000° F. of austenite containing chromium and ferrite containing nickel.

2. *Examination for Grain Orientation and Effects of Mechanical and Heat Treatment:*

The incidental use of X-rays in determining the orientation of single metal crystallin wires and rods prior to mechanical and physical tests has been advocated and extended by McKeehan[6] in his studies of magnetism, twinning, etc. Thus far, most of the theoretical and experimental work on the mechanism of plastic deformation and recrystallization of pure metals as deduced from X-ray results has been conducted in Germany, England, and Japan. Much of this work has been tested in the United States[1] with the following conclusions concerning the direction of the fibering or deformation axis:

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[1] *Proceedings*, Royal Society, 112A, 678 (1926).

[2] *Transactions*, Am. Soc. Steel Treating, Vol. 9, p. 717 (1926).

[3] *Nature*, Vol. 119, p. 494 (1927).

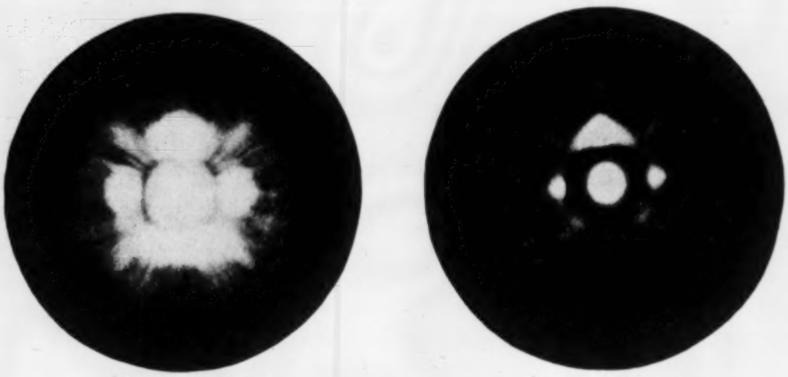
[4] Am. Inst. Mining and Metallurgical Eng., No. 1657-E (1927).

[5] Am. Inst. Mining and Metallurgical Eng., No. 1850-C (1927).

[6] *Nature*, Vol. 119, p. 705 (1927).

	DRAWING	ROLLING
Face-centered cubic metals	[111] [100]	[335] nearly [111]
Body-centered cubic metals	[110]	[110]

Reference should be made to the remarkable new results on recrystallization which show the persistence of preferred orientation after annealing, and the effects of small amounts of impurities. Copper when first hot rolled, then cold rolled and finally strongly annealed is characterized by orientation of cube edges in the direction of rolling, so that a sheet is obtained acting essentially like a single crystal and with mechanical properties altogether different from a sheet of small randomly oriented grains. X-ray studies have proved that 0.05 per cent of iron in silver has a very great effect on recrystallization in preferred directions at room temperature and thus deleteriously affects silver plate unless copper is also present as impurity. By such fundamental studies the fabrication of metals is being reduced to a science.[2] Every impetus should be given to researches of this type in America.



(*a*) Hot-Rolled. (*b*) Cold-Rolled.

FIG. 27.—Imperfect Fiber Structures of Low-Carbon-Steel, Showing Asymmetry Characteristics of Direction of Last Pass Through Rolls.

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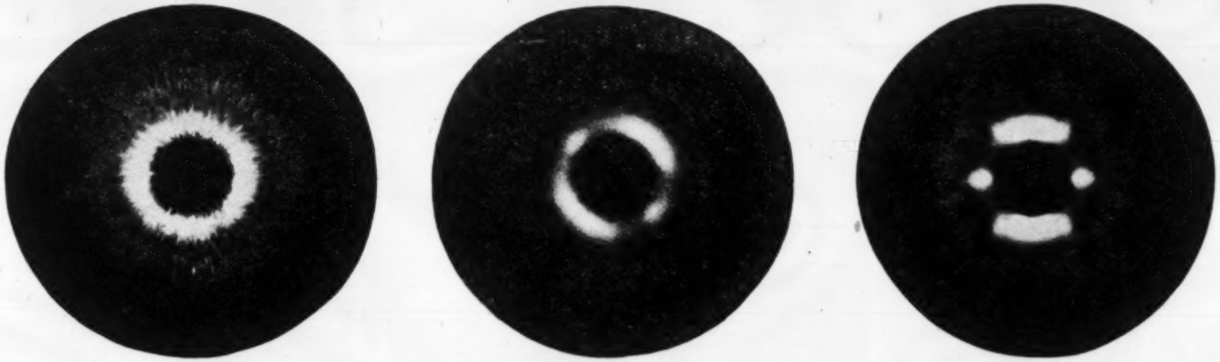
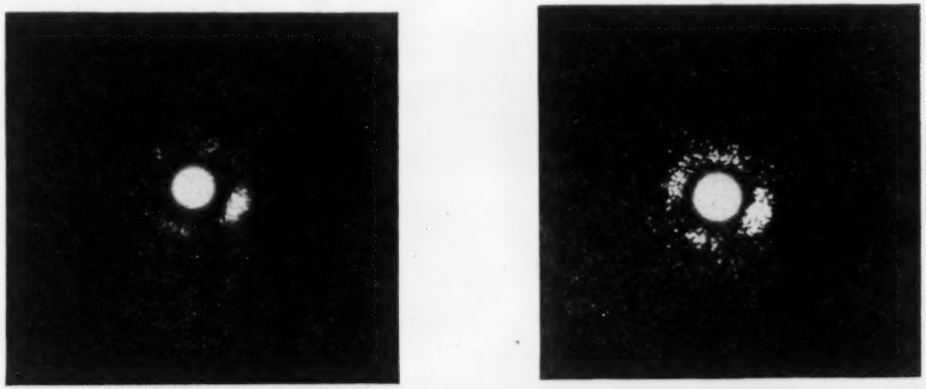
[1] For example, Norton and Warren, Am. Inst. Mining and Metallurgical Eng., No. 1610-E (1927).

[2] For the most recent work see Glocker, *Zeitschrift Physik*, Vol. 31, p. 386 (1926); Schmid and Wasserman, *Zeitschrift Physik*, Vol. 40, p. 451 (1926); Glocker and Widmann, *Zeitschrift Metallkunde*, Vol. 19, p. 41 (1927); Sachs *et al.*, *Zeitschrift Physik*, Vol. 41, p. 889 (1927); Vol. 42, p. 283 (1927).



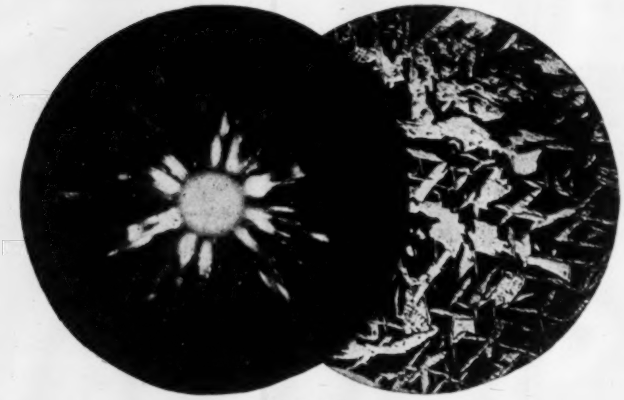
(*a*) Original Bar. (*b*) Intermediate Reduction. (*c*) Final Rolled Sheet.

FIG. 28.—Change in Pattern with Reduction by Rolling of Nickel Ingot.

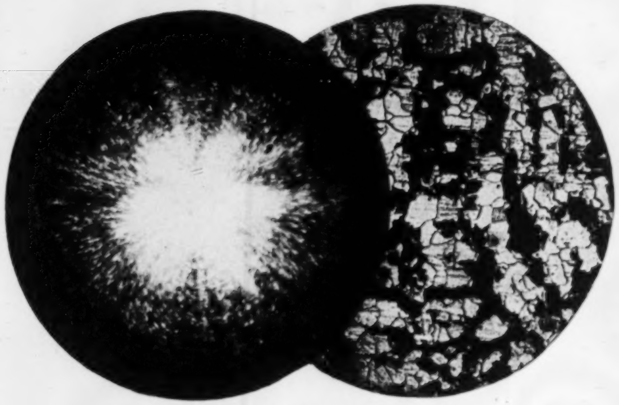


(*a*) (*b*)

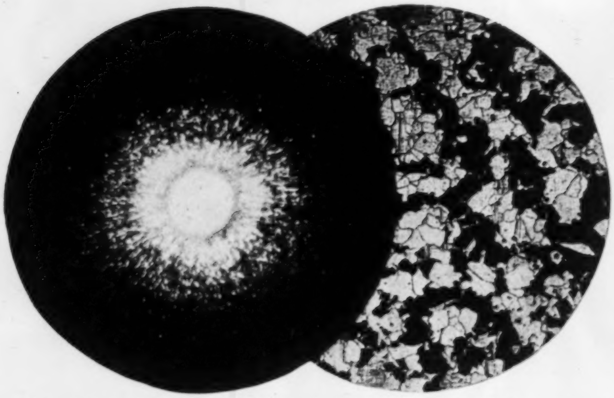
FIG. 29.—Variation of Structure in the Same Sheet of Annealed Sheet Steel.



(a) As Cast.



(*b*) After 5-hour Anneal.



(*c*) After four 5-hour Anneals, Showing Ideal Structure.

FIG. 30.—Diffraction Patterns and Photomicrographs of Cast Steel, Illustrating Value of X-ray Method in Controlling Heat Treatment.

IV. PRACTICAL APPLICATIONS OF THE PINHOLE DIFFRACTION METHOD TO THE STUDY OF COMMERCIAL METALS

1. *Effects of Fabrication of Metals:*

Regarding the general evidences of mechanical and of heat-treatments, of even greater commercial importance than the determination of constitution of metals and alloys by X-rays is the detection of the effects of any kind of fabricating process upon metals in terms of structure and of practical behavior. The photographs presented herewith illustrate in general the possibilities of the type of examination.

The two views in Fig. 27 show the diffraction patterns obtained with ordinary low-carbon sheet-steel after hot and after cold rolling. Both show the characteristic fiber evidences, which indicate that these metals must have directional properties. The cold working produces more of the fibering than does the hot rolling. Both processes evidence radial streaks that are indicative of distortion strain. The lack of symmetry in the diagram results from the fact that the direction of last pass through the rolls determines the structure and the diagrams, which correspond to a rolling direction from bottom to top.

Figure 28 shows three stages in the rolling of nickel sheet, the first for the ingot bar, the third for the final product and the second for intermediate reduction.

Subsequent heat-treatment, of course, relieves both the strain and the directional properties. The X-ray method is extremely sensitive to this process, as is illustrated by the two patterns in Fig. 29 taken at different positions of the same sheet.

2. *Cast Steel:*

Although it is commonly thought that cast metals generally exhibit a random orientation of grains, an examination of Fig. 30 will show the great practical importance of the pinhole method in this field. The coarse non-uniform grain mass of steel as ordinarily cast, shown at the top of Fig. 30, is by no means obliterated in the standard annealing processes, as the middle view of Fig. 30 shows. The removal of internal stresses is not impossible under proper annealing conditions. The bottom view of Fig. 30, for the same casting, shows the ideal structure of small equi-axed crystals with neither directed orientation or intensity maxima nor internal strain or radial asterism striation. Physical data are as follows:

	BRINELL HARDNESS NUMBER (500-KG. LOAD)	ROCKWELL HARDNESS NUMBER
As cast	136	89
5-hour anneal	126	85
Four 5-hour anneals	**135**	**87**

The grain refinement in the last case is caused by successive passages through the critical point.

A casting of irregular form, radiographically perfect in freedom from holes, cracks and inclusions, was examined by the Laue pinhole method to ascertain the uniformity of structure and the distribution of strain. In the same casting, depending upon the thickness of the cross-section and the zone relative to the mold or core wall, the diagrams varied as widely as those at the top and bottom of Fig. 30. Upon the basis of the X-ray photographs alone, it was possible to plot regions of equal strain or "isostrainal" lines and to predict how and where the casting would fail and crack. Upon the basis of these facts it was thus possible to introduce modifications in the casting technique, particularly in the annealing, following each change with

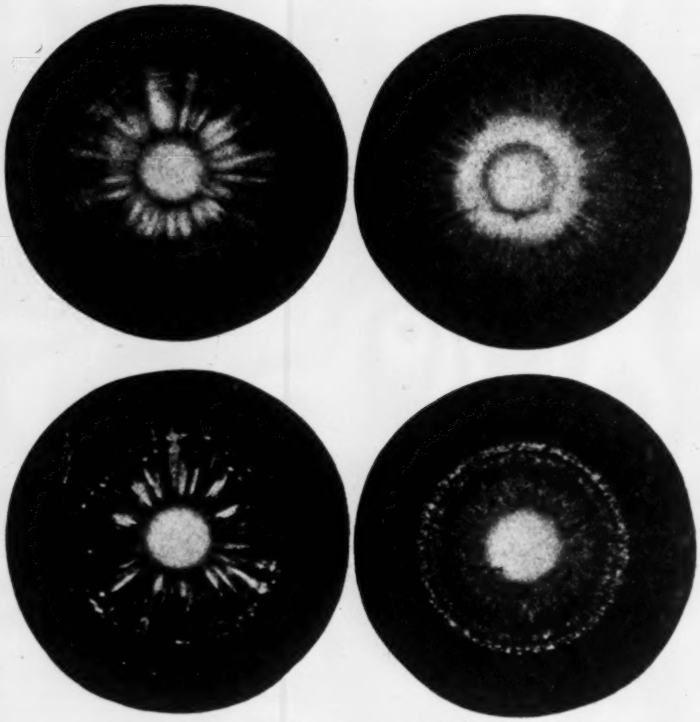


FIG. 31.—X-ray Determination of Weld Structure.

The X-ray structure of a lap weld obtained by the ordinary arc-method in air is shown in the two upper views. The structure as obtained by the General Electric Company method of welding in hydrogen is shown in the two lower views. For each pair, the structure in the weld is shown at the left, and that of the metal adjacent to the weld is shown at the right. The variations in particle size and strains in the various zones between the weld metal and the original bar-metal were clearly disclosed by the X-ray photographs.

the X-ray diagrams and thus arriving at castings that were far superior in uniformity of structure. In the field of casting, therefore, this represents a great advance in research beyond the point of the radiographed casting. The information pertains to the fine structure and to the phenomena not otherwise observable.

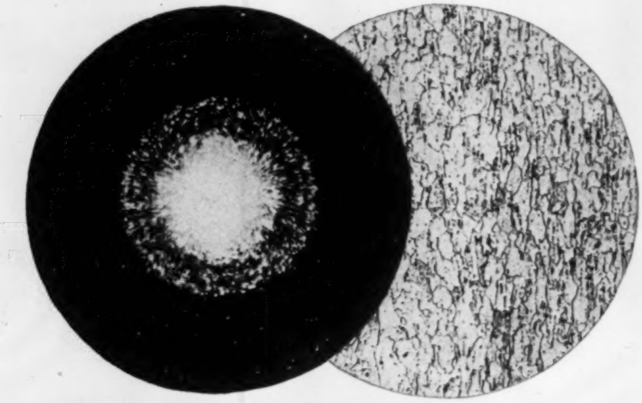
In agreement with the facts presented in the preceding section of this paper, the last traces of asterism or strain in steel castings are remarkably persistent. In a comprehensive study of the effects of annealing upon fine structure it was found that, for blocks only ½ in. thick, heating for 20 hours at 900° C. (1652° F.) was necessary for removal of the last traces of inhomogeneity, although improvement was observed successively at intermediate times. Changes in the particle size and strain during annealing and the effects of the method of cooling, either slowly in the furnace or by quenching in air, water and oil, were all easily determined by the X-ray diagram.

3. *Welds:*

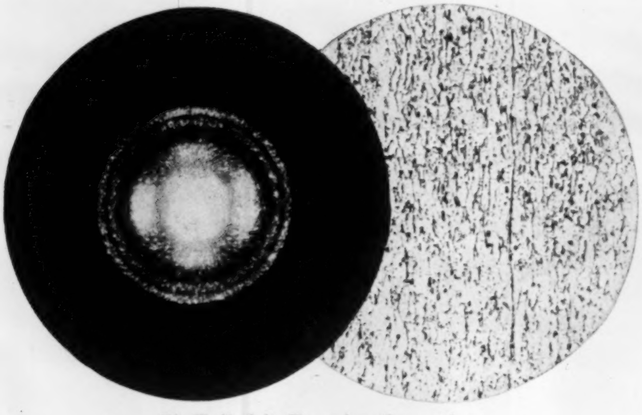
The determination of the structure of welds from the X-ray diffraction diagram has been very illuminating, particularly when these results are combined with photomicrographs and tensile-strength tests. The specimens were various butt and lap welds produced by the arc method in air, shown in the upper pair of Fig. 31 and by the General Electric Company's method of welding in hydrogen, shown in the lower pair. The variations in particle size and strains in the various zones between the weld metal and the original bar-metal, were clearly disclosed by the X-ray photographs. The maximum strain as judged by asterism is observed in the top of the weld metal, where the last metal has been added and the cooling has been most rapid; while the smallest particle size is in the refined zone of the original metal adjoining the weld. The hydrogen-atmosphere welds are distinguished from the ordinary type by larger grain-size but a far more uniform grain-size throughout all the zones, even in the refined zone, and a much greater freedom from strain, as if the composite structure were annealed. The oxide impurities at grain boundaries in the ordinary welds are clearly absent in the hydrogen welds so that there is no interference with grain growth. In spite of large grains in random orientation, these welds are fairly ductile, illustrating an important metallurgical fact that large grains do not necessarily mean brittleness of the specimen. Further improvements undoubtedly will result in a still more satisfactory weld, and X-rays may play a primary rôle in such improvements that will lead to new standards and new specifications in the numerous instances in which welds are necessary.

4. Forming Steel:

The prime requisite of a good forming steel such as is used in stamping operations like those for automobile radiators is obviously an absence of any directional properties; hence, the grains should be equi-axed and well knit together without strain or fibering. One of the great metallurgical problems at present is a satisfactory test or method of prediction of the behavior of steels in forming; photomicrographs and physical tests may often be identical for two sheets, and yet one will crack in forming while the other easily withstands the strain.



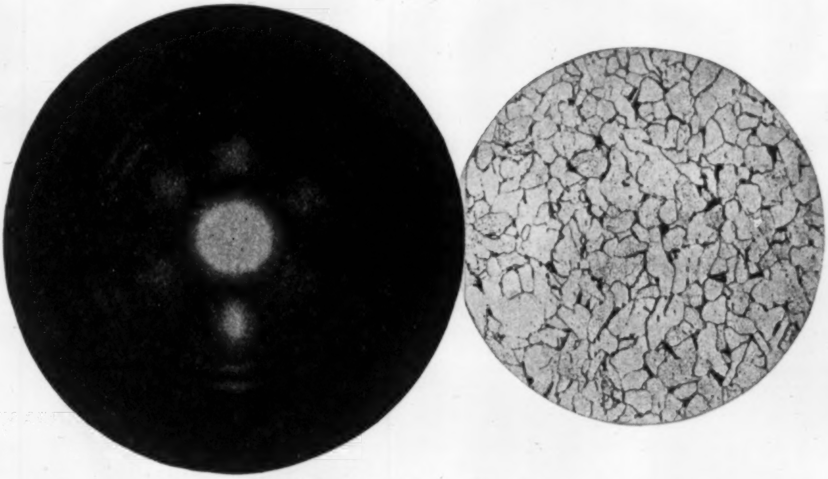
(*a*) Formed Satisfactorily.



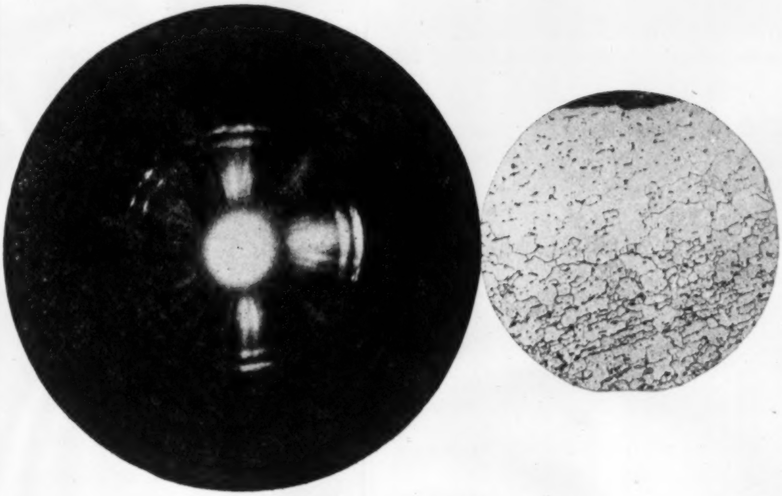
(*b*) Failed in Forming Operation.

FIG. 32.—Diffraction Patterns and Photomicrographs of Forming Steels.

Figure 32 is a reproduction of the X-ray diffraction patterns of such a pair of sheets, the good sheet being shown at the top and the bad one at the bottom. The difference is unmistakable; the sheet that fails invariably shows strong evidence of a residual preferred orientation and strain and often shows an unsymmetrical and distorted pattern. The satisfactory sample is distinguished by a pattern approaching the continuous Debye-Scherrer rings of a random aggregate which thus has the same properties in all directions.

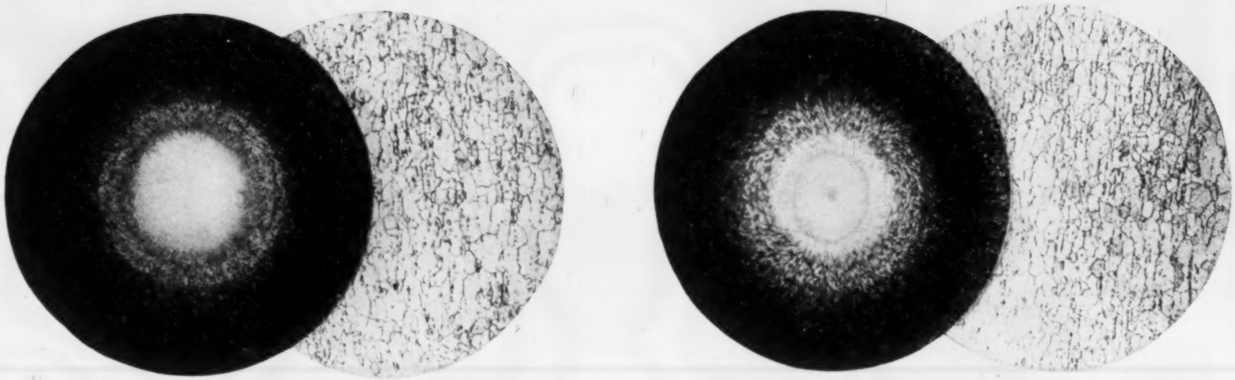


(*a*) After Satisfactory Forming.



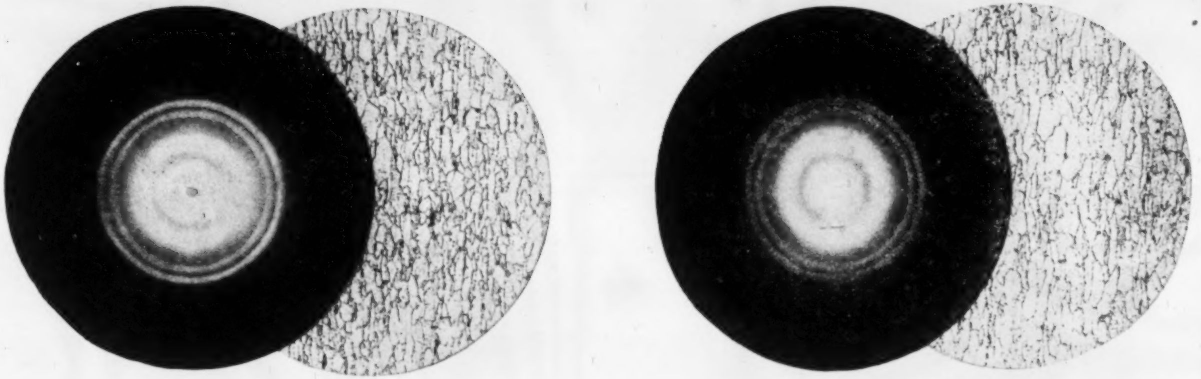
(*b*) After Unsatisfactory Forming.
Note Non-Uniform Pattern and Structure.

FIG. 33.—Structure of Forming Steel.



(*a*) Soft.

(*b*) Quarter Hard.



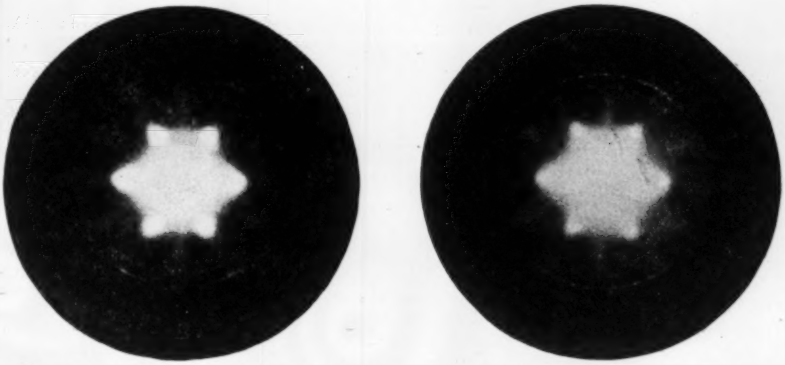
(*c*) Half Hard.

(*d*) Hard.

FIG. 34.—Commercial Grades of Forming Steels.

The two views of Figs. 33(*a*) and (*b*) show the structures of sheet steel after forming, whether satisfactory or unsatisfactory. A very sensitive method is required because the photomicrographs may appear identical. The development of a technique of manufacture and particularly of annealing practice which will reduce rolling strain to the minimum and assure uniformly good forming-steel might well be made upon the basis of the X-ray method.

In Fig. 34 is an interesting and important series of diffraction patterns for steels, of the same composition, technically classed and marketed as soft, quarter-hard, half-hard, and hard, depending upon the treatment. The point of particular interest is the very great change in structure between quarter and half-hard as compared with the other transitions. The corresponding physical data (averages of several specimen determinations) are as follows:

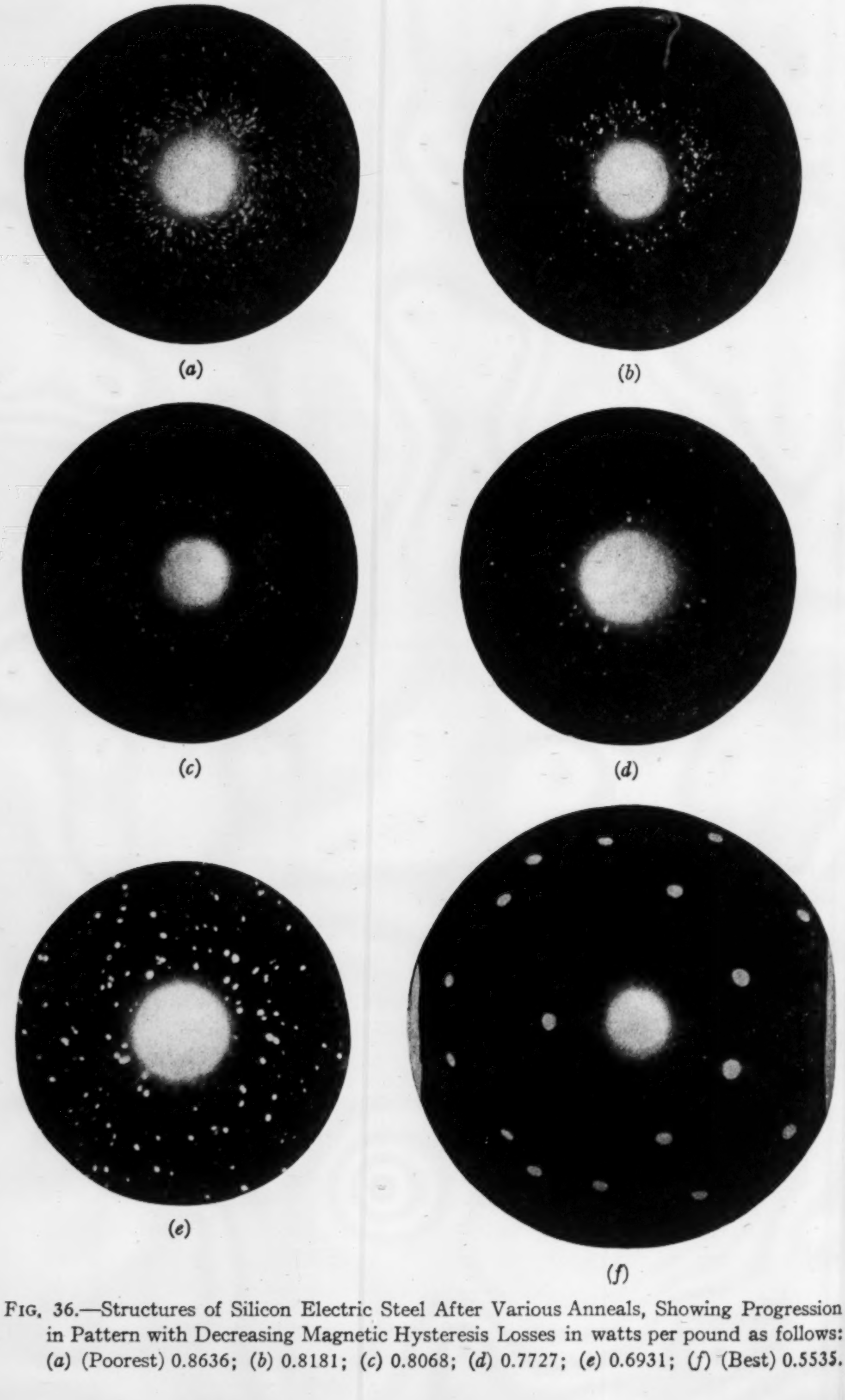


(*a*) Unsatisfactory and Cut Die. (*b*) Drawn Satisfactorily.

FIG. 35.—Structures of Steel Wire After Intermediate Anneal.

Grade	Thickness, in.	Erichsen Pressure, lb.	Depth of Cup, in.	Scleroscope
Soft	0.033	4200	0.430	15
	0.031	4200	0.420	
Quarter-hard	0.031	4100	0.423	15
	0.031	4100	0.421	
Half-hard	0.031	3000	0.276	33
	0.031	3400	0.270	
Hard	0.031	3300	0.238	35
	0.031	3300	0.238	

Another series of sheets 0.010 in. thick have been studied; the X-ray patterns correspond to those of the thicker sheets when moved back one grade (that is, half-hard (thin) = quarter-hard (thick)).



(*a*) (*b*)

(*c*) (*d*)

(*e*) (*f*)

FIG. 36.—Structures of Silicon Electric Steel After Various Anneals, Showing Progression in Pattern with Decreasing Magnetic Hysteresis Losses in watts per pound as follows: (*a*) (Poorest) 0.8636; (*b*) 0.8181; (*c*) 0.8068; (*d*) 0.7727; (*e*) 0.6931; (*f*) (Best) 0.5535.

5. Steel Wire:

A difficult metallurgical problem has been to determine the cause of the cutting of dies during the drawing of steel wire. One explanation has been that the wire is too hard in some parts; another has been that there are soft spots. Figure 35 shows the structures of partly finished low-carbon-steel wire after an intermediate anneal; the wire represented in the right-hand view gave a satisfactory subsequent drawing and that on the left an unsatisfactory, or over-size, drawing. The evidence is convincing that the unsatisfactory wire was improperly annealed and was too hard, since the fibering is still strong and the grain size is small as compared with the properly annealed sample having the structure shown on the right.

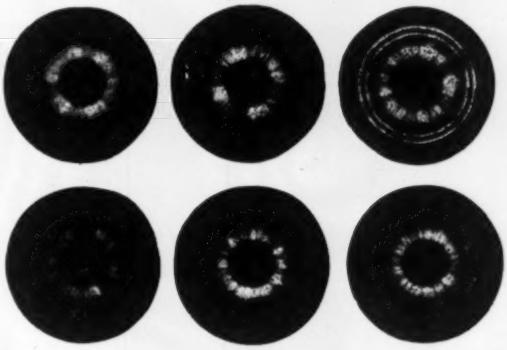
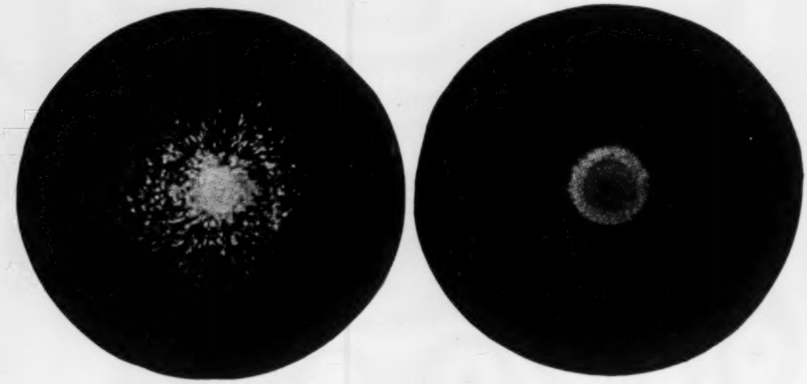


FIG. 37.—Patterns in Various Parts of Steel Rail with Transverse Fissure (Lower Right Shows Structure of Normal Steel).

6. Electrical Steel:

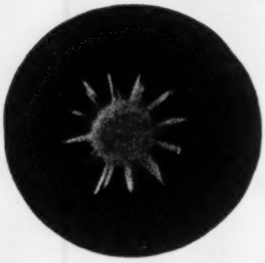
In reference to steel suitable for electrical construction, one of the most eloquent examples of the value of X-ray diffraction research in metallurgy is in the field of the steels used as cores of transformers and the like where the magnetic properties are the chief concern. These metals usually contain 3 to 4 per cent of silicon, together with carbon and the usual impurities. It has long been known that large grain size is an important factor, but specifications usually are based upon the magnetic hysteresis loss in watts per pound. Figure 36 shows the diffraction patterns of a series of silicon-steel samples, differing essentially in the annealing only. The upper left view indicates a partial or unsatisfactory anneal showing a high hysteresis loss of 0.8636 watts per pound; the lower right, a controlled anneal showing a low hysteresis loss of 0.5535 watts per pound; the others show progression in pattern and hysteresis losses between the limits.

It is at once apparent that, as the hysteresis losses decrease, the diffraction spots become larger and fewer in number, culminating for minimum loss and best magnetic properties in the diagram of a single-crystal grain extending through the sheet, as indicated in the lower right view. The faulty annealing of the poorest specimen is further shown by the four clustered intensity maxima symmetrically placed around the direct-beam spot; these are residual evidence of the fibering originally produced by the rolling of the sheets before heat-treatment. The X-ray pattern alone may serve, therefore, as evidence of the magnetic properties of the silicon steel, for the essential attribute is the minimum number of unstrained single grains; and this number is proportional roughly to the number of spots on the X-ray pattern.



(*a*) Ordinary deposit.

(*b*) Deposit from solution containing 0.5 per cent gelatin.



(*c*) Lead dendrite.

FIG. 38.—Structures of Electrodeposited Lead.

7. *Rail Steel:*

Researches are now under way on the structure of steel rail as it is involved in transverse fissures. In Fig. 37 are assembled several of the first photographs of a defective rail. A normal rail shows a perfectly uniform inner diffraction ring, while the defective structure shows streaks and even evidence of fibering.

8. Electrodeposited Metals:

Following the earlier studies of Clark and Frölich on electrodeposited nickel, Frölich, Clark and Aborn[1] have made an X-ray investigation of lead electrodeposited under a very wide range of conditions, and have thus discovered the method of producing the most uniform and firmly adhering films for commercial purposes. These studies demonstrated the structure of lead dendrites, the orientation of grain on the electrode, and the remarkable effects of traces of gelatin and related addition agents in reducing grain size (Fig. 38).

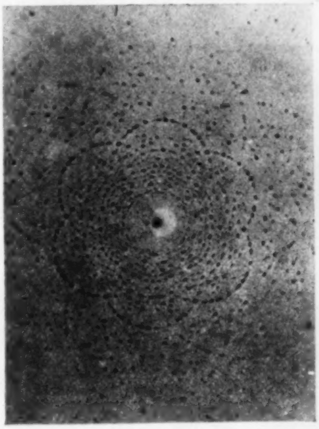


FIG. 39.—Laue Pattern of the Mineral Beryl.

V. CRYSTALLIN INORGANIC MATERIALS

Most of the work on inorganic substances has been devoted to the investigation of the unique crystal structures of more than 350 pure compounds. Some of these are very complex and the interpretation of X-ray data involves ingenuity of the highest order. In Fig. 39 is shown the Laue pattern for a crystal of the mineral beryl $Be_3Al_2Si_6O_8$, which W. L. Bragg was able to interpret completely. Industrially important inorganic materials may be studied from the practical point of view, only a few of which will be mentioned. Miss Farnsworth, working in the author's laboratory, has made an extended X-ray study of limes having different plasticities.[1] The calcium oxide samples which give plastic hydrates produce only the diffraction pattern for pure CaO, and the plastic hydrates give an hexagonal pattern with axial rates 1.40. The patterns of the less plastic samples are complicated by additional lines of $Ca(OH)_2$ in the first case, and of $Ca(OH)_2$ and $CaCO_3$ in the second, the intensity of these lines being a measure, inversely, of plasticity. High temperatures and vacuum burning are conducive to greater plasticity. Azbe[2] has applied the results of X-ray crystal structure studies to the practical problems of lime-burning in a manner which promises much for further applications of X-rays in the industry. At the Bureau of Standards[3] portland cement in all its phases is being subjected to X-ray investigation starting with the compounds CaO, $CaCO_3$, SiO_2, Al_2O_3, Fe_2O_3, $Ca(OH)_2$, $3CaO \cdot Al_2O_3$ and $5CaO \cdot 3Al_2O_3$.

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[1] *Transactions*, Am. Electrochemical Soc., Preprint 1926; *Zeitschrift Electrochem*, Vol. 32, p. 295 (1926).

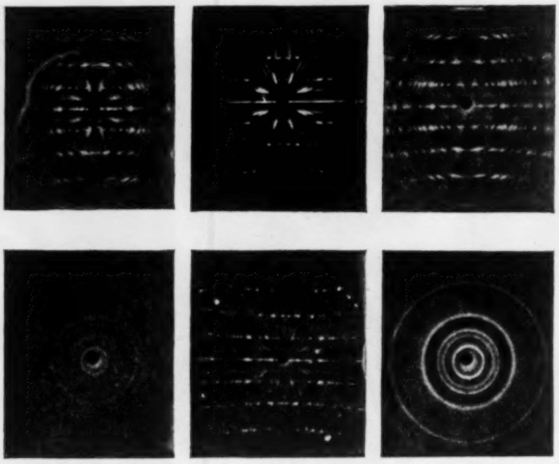

FIG. 40.—Patterns for Six Types of Asbestos (See also Fig. 17).

Bozorth[4] has analyzed with X-rays the Bower-Barff protective coating, produced by the action of steam on iron at 700° F. with subsequent cooling in air, and identified layers of ferrous oxide, magnetite and ferric oxide in order, with respective thicknesses of 10^{-2}, 2×10^{-4} and 2×10^{-5} cm.

The studies of Wyckoff and Morey[5] on the system soda-lime-silica parallel in one sense the studies of the ceramic materials mullite and sillimanite which gave practically identical patterns. Thus, Na_2CaSiO_4, $Na_4Ca(SiO_3)_3$ and $Na_2Ca_2(SiO_3)_3$ all give very similar patterns in which the strong lines all coincide, while the differences lie in additional weak lines.

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[1] *Industrial and Engineering Chemistry*, Vol. 19, p. 583 (1927).

[2] *Industrial and Engineering Chemistry*, Vol. 19, p. 600 (1927).

[3] *Am. Journal of Science* 13, 467 (1927).

[4] *Journal*, Am. Chemical Soc., Vol. 49, p. 969 (1927).

[5] *Am. Journal of Science*, Vol. 12, p. 419 (1926).

There is a growing application of X-rays to the identification of minerals. Clark, Aborn, and Brugmann[1] announce, preliminary to detailed publication, the remarkable differences in the X-ray patterns from various types of asbestos, and the changes in these as the asbestos is chemically treated (acid, heat, etc.). In Figs. 17 and 40 are shown the patterns from 7 varieties of asbestos. Only one kind of asbestos is suitable as a base for catalysts and its purchase for this purpose may be made upon the basis of a single X-ray test diffraction photograph. (Fig. 17.)

Miss Farnsworth[2] has applied the X-ray method also to investigation of the cause of loss of tensile strength of plaster-of-paris molds after recalcination. The particle size indicated in these patterns was definitely larger the smaller the tensile strength. The addition of 0.25 per cent of Al_2O_3 prevented increase in particle size with successive reheatings and thus, also, the decrease in tensile strength.

VI. CRYSTALLIN ORGANIC COMPOUNDS

Most of the pioneer work in the field of applying X-ray diffraction methods to organic chemistry has been carried on in the laboratories of Bragg in England and deBroglie in France. These have been along the lines of unique analyses of single crystals, the interpretation of stereoisomerism, molecular symmetry, optical activity, the orientation, length, and polymorphic forms of long chain compounds in thin films, and the analysis of natural materials such as waxes, greases, butters, etc.

The outstanding result of X-ray studies of long-chain compounds is that the lengths of molecules may be measured, inasmuch as in thin films the molecules arranged themselves parallel to each other in successive diffraction layers, to which the molecules are more or less perpendicular. Complications arise from the fact that some pure substances may show as many as three different spacings. In general, however, the long diffracting spacings in an homologous series of paraffin hydrocarbons, acids, alcohols, soaps, esters, ketones, glycerides, etc., are linearly related to the number of carbon atoms. All experimental data to date are plotted in Fig. 41. The experimenter may thus analyze any unknown mixture of long carbon chains.

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[1] *Journal*, Soc. of Automotive Engrs., Vol. 20, p. 291 (1927).

[2] *Industrial and Engineering Chemistry*, Vol. 19, p. 714 (1927).

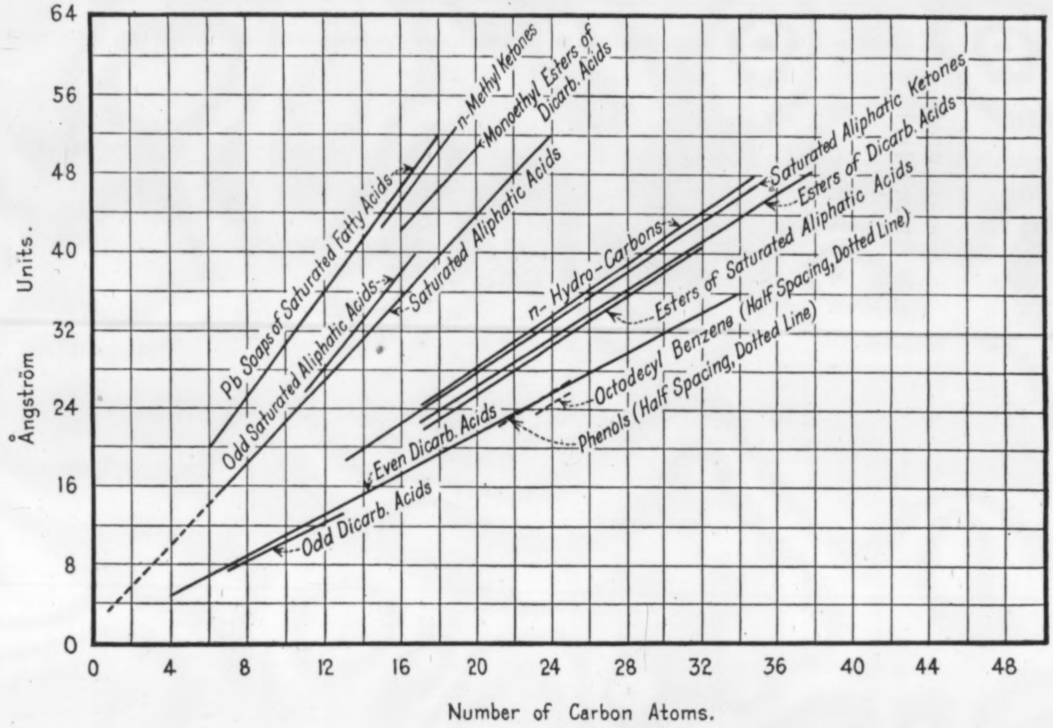

FIG. 41.—Graphical Relation of the Principal Diffraction Spacings for Long-Chain Compounds, in Ångström Units *versus* Number of Carbon Atoms.

Clark[1] has studied paraffin waxes and observed that these mixtures of as many as 18 hydrocarbons give diffraction lines for only one hydrocarbon. The spacings corresponding to chain lengths vary depending upon the rate of cooling, and impurities for any given commercial wax. Such properties as transparency are related to these spacings. Clark, Aborn and Brugmann[2] have determined the conditions which are advantageous for molecular orientation of long-chain compounds (nature of backing, thickness of film, time in fluid state) and applied these observations to the case of lubricants. In Fig. 42 is shown the diagram for a lubricant worked as in a bearing. The fibering, already familiar in the case of metals, is clearly apparent.

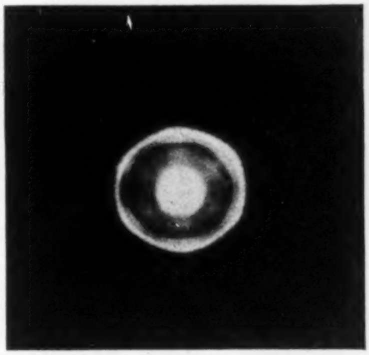

FIG. 42.—Diagram for Grease Worked as in a Bearing, Showing Fibering Orientation of Molecules.

The soaps and glycerides in linseed and tung (china wood) oils have been studied also.

Another interesting outgrowth of organic crystal analyses is the application of some of the long-chain compounds with large spacings as crystal gratings for the measurement of very long wave lengths. In this way Dauvillier in France[3] was able to bridge the spectral gap from 20 to 136 Å using lead melissate (spacing 87.5 Å) as grating.

VII. COLLOIDAL AND AMORPHOUS MATERIALS

1. *Catalysts:*

Clark[4] in continuing work on particle size of metal catalysts as related to the activity, finds from photometric measurements of line breadths that there is a definite relationship between decreasing particle size to an optimum size, with increasing activity. These investigations of catalysts are being continued in order to discover the effect of every possible variable. The earlier work on nickel catalysts has inspired the new and excellent X-ray studies of catalysts by Levi in Italy.

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[1] *Nature*, Vol. 120, p. 12 (1927).

[2] *Journal*, Soc. of Automotive Engrs., Vol. 20, p. 291 (1927).

[3] *Journal Physique et la Radium*, Vol. 8, p. 1 (1927).

[4] Colloid Symposium Monograph, Vol. IV, p. 157 (1926).

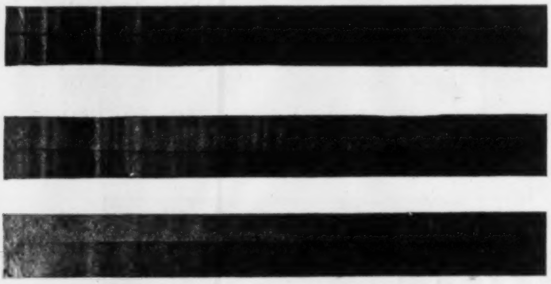


FIG. 43.—Spectra of Samples of Tin Dioxide Showing Effect of Decreasing Particle Size in Widening Lines.

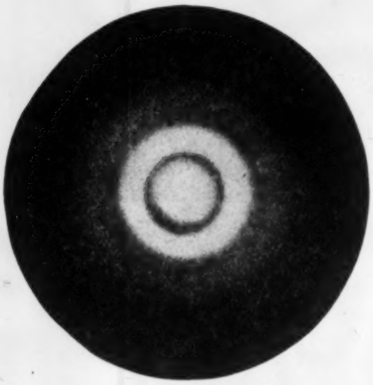


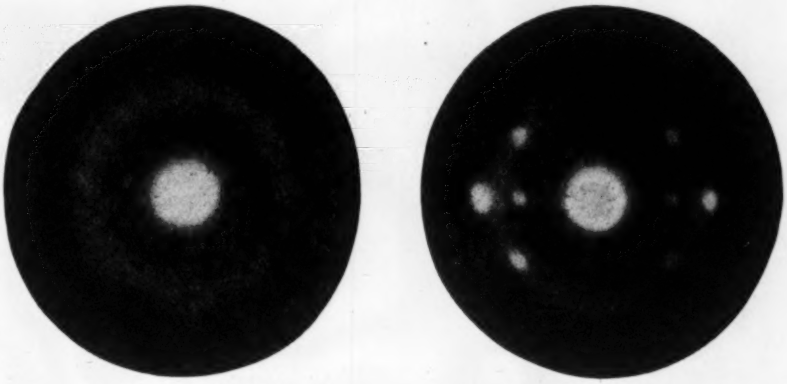
FIG. 44.—Pattern for Oil-Quenched Case-Hardened Steel, Showing Minute Grain Size and Distortion.

2. Practical Studies of Colloidal Particle Size:

The above type of investigation can be applied with great value to the study of paints, pigments, and enamels. Clark, in the paper just referred to, reports measurements on a series of samples of stannic oxide used in enamels in which the particle size varied from 10^{-8} to 5×10^{-7} cm. as determined from the widths of the diffraction lines (Fig. 43). Obviously, covering and obscuring power are intimately related to particle size. Clark and Brugmann measured the particle size of case-hardened steel (martensite and troostite) to be nearly 10^{-7} cm. (Fig. 44).

3. Carbon Black:

A continuous transition by heat treatment between an active charcoal with an X-ray pattern indicating as nearly the amorphous state as possible (one very ill-defined ring) and graphite (sharp, many-line crystal spectrum) has been observed by Clark and Alm.[1] They found that the change in X-ray pattern of the active carbon, lags behind the change in physical and chemical properties to those of graphite. This is evidence of a paracrystallin state in which crystallin layers are still too few and distorted to produce definite interference maxima. These studies are promising for the discovery of the desired properties of carbon in rubber.



(*a*) Unstretched (Amorphous) Rubber. (*b*) Stretched (Crystal) Fiber.

FIG. 45.—Patterns for Unstretched (Amorphous) and Stretched (Crystal-Fiber) Rubber.

4. Rubber and Allied Substances:

The first published account of the fact that rubber when stretched produces a crystallin fiber diffraction pattern was by Katz. This fact had been observed by Clark; the essential phenomena described by Katz, though not the details, have been confirmed by him[2] and by Hauser and Mark in Germany. Clark and Hauser and Mark agree that a unit cell containing $(C_5H_8)_8$ is indicated from X-ray data, and that upon stretching, a lower polymer phase is forced out of a higher polymerized phase which is thereupon oriented for diffraction. Figure 45 shows the amorphous pattern for unstretched rubber, and the fiber-crystal structure of stretched rubber. That this is due to the rubber hydrocarbon is demonstrated by new work by Clark on very pure soluble rubber. The most interesting feature at present is that no sample of synthetic rubber has been found to produce this sharply fiber diagram. It is possible that this is the criterion of a successful artificial reproduction of rubber. Another recent development is the X-ray study of "racked" rubber, stretched 10,000 per cent. Whereas rubber stretched once begins to show a fiber pattern at 75 per cent elongation, "racked" rubber when released with a permanent set of 1000 per cent produces only the amorphous pattern. Clark has reported the only results and calculations on balata and gutta percha which differ from each other and from rubber;

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[1] Colloid Symposium Monograph, Vol. IV, p. 155 (1927).

[2] *Industrial and Engineering Chemistry*, Vol. 18, p. 1131 (1926).

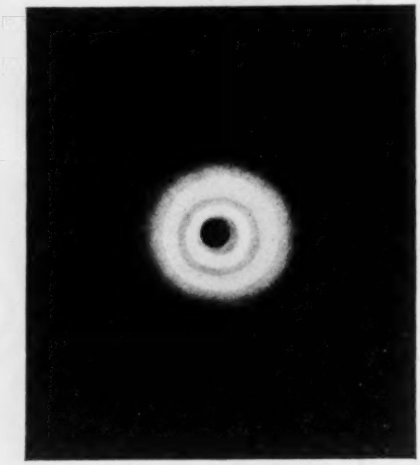

FIG. 46.—Typical Pattern for Amorphous Material (Liquids, Gels, etc.): Cumar Varnish.

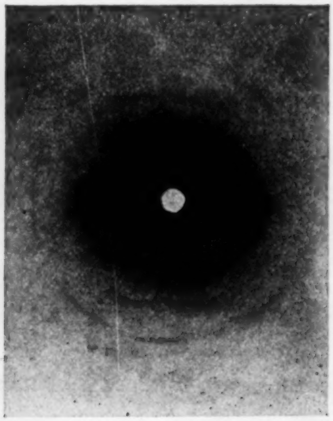

FIG. 47.—Crystal-Fiber Structure of Pine Wood.

both contain crystallin matter when unstretched while rubber ordinarily does not. Gelatin and glue in stretched condition also show an incipient crystal-like arrangement. The polymerization of shellac upon heating has been followed by X-ray photographs.

5. *Liquids and Gels:*

Great interest has been aroused recently in the interpretation of "amorphous" patterns consisting of two or three diffuse diffraction rings. Figure 46 shows such a pattern for cumar, used as varnish. It is now definitely established that these are indicative of the distance of nearest approach of molecules or aggregates acting as units. Stewart and Morrow[1] propose the term cybotaxis for this state, and they propose to apply the conception to osmosis and other phenomena. They present results on liquid primary normal alcohols which show two spacings, one corresponding to chain length and one (constant for all) to cross-section in every way analogous to results with the solid state. Clark, Aborn, Brugmann, and Davidson[1] have discovered that polymerization, oxidation, photochemical action, and solvent swelling of gels in rubber, nitro-cellulose, linseed oil, and tung (china wood) oil (liquid and dried films) are indicated in a remarkable way by these simple patterns. In the last two cases, over-aging is accompanied by actual crystallization. Representative data are given in the following table:

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[1] *Proceedings*, National Academy of Science, Vol. 13, p. 222 (1927).

DIFFRACTION SPACINGS FOR NITRO-CELLULOSE

		FRESH	LIGHT AGED	HEAT AGED
Untreated dry sheet..........	inner	7.18 Å	7.17 Å	6.98 Å
	outer	4.02 Å	4.05 Å	3.92 Å
Solvent softener.............	inner	9.30 Å	9.32 Å	7.86 Å
	outer	4.47 Å	4.47 Å	4.31 Å
Oil softener.................	inner	7.34 Å	7.50 Å	7.50 Å
	outer	4.31 Å	4.39 Å	4.39 Å

DIFFRACTION SPACINGS FOR TUNG (CHINA WOOD) OIL

	INNER RING	OUTER RING
Raw liquid.......................	8.5 Å	4.4 Å
Raw dry film (oxidation at room temperature).................	+ 6 per cent	+ 1 per cent
Raw gel (polymerized by heat).....	+ 3 per cent	+ 1 per cent
Liquid prepared with turpentine....	+12 per cent	+16 per cent
Pseudo-crystallin dry film..........	+13 per cent	+ 6 per cent

6. Cellulose and Silk:

Clark, Freudenthal and Kulp[2] have subjected a long series of natural cellulose fibers and all types of rayon to X-ray investigation and found them all crystallin except diacetate rayon. Figure 47 is the pattern for pine wood, and Fig. 48 for a ramie fiber. For natural or regenerated cellulose the same lattice as found by Herzog, Katz, Sponsler and Dore, and others is indicated (rhombic $(C_6H_{10}O_5)_4$), but the degree of fibering varies widely from random in a viscose film to nearly perfect in ramie and flax. Tension on the fiber in rayon production has large effects upon the fibering as disclosed by X-ray

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[1] *Proceedings*, National Academy of Science, July, 1927.

[2] *Proceedings*, Am. Assn. Textile Chemists and Colorists, 1926, p. 234; *Am. Dyestuff Reporter*, Vol. 15, p. 788 (1926); *Proceedings*, Technical Assn. Pulp and Paper Industry, 1927; *Paper Trade Journal*, Vol. 84, p. 47 (1927); Colloid Symposium Monograph, Vol. IV, p. 169.

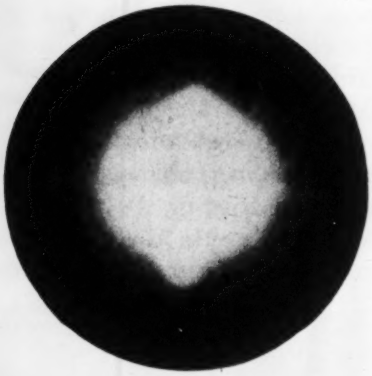

FIG. 48.—Diffraction Pattern of Ramie Fiber.

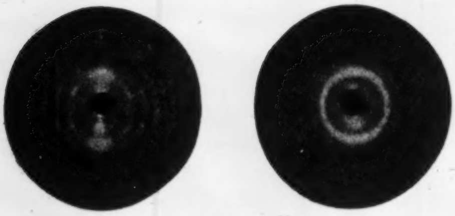

FIG. 49.—Diagrams Showing Extremes in Fibering of Same Type Rayon.

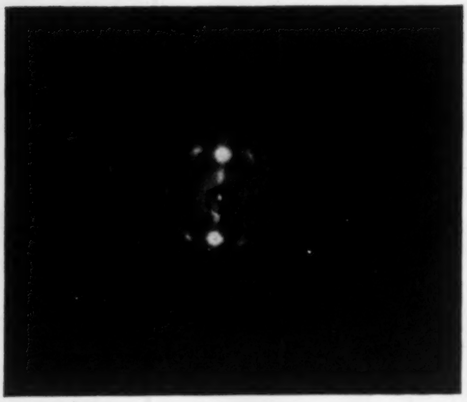

FIG. 50.—Fiber Diagram of Natural Silk.

diffraction and also upon important practical properties. Figure 49 shows two extremes of the same rayon, one in which the fiber structure is strong, indicating regular arrangement of the crystal units with respect to the fiber, and the other a nearly random arrangement. The latter specimen did not compare with the former in strength and durability. By way of comparison the sharply fibrous structure of the natural protein silk is shown in Fig. 50. In every phase of the textile industry, from raw material to finished cloth, such as washing, swelling, drying, mercerization, dyeing, aging, etc., the X-ray diffraction method may be used to good advantage.

Thus, in 1927 do we dedicate the present achievements and the future promises of a new, and certainly the most fundamental science of examination of materials to that spirit of eager progress in the American Society for Testing Materials and in industry so well exemplified by Edgar Marburg.

EMBRITTLEMENT OF BOILER PLATE[1]

S. W. Parr[2] and Frederick G. Straub[3]

Synopsis

The term "embrittlement of boiler plate" is defined as the inter-crystallin cracking in the riveted areas. This difficulty is confined entirely to a type of water supply or water treatment which brings about a predominance of sodium carbonate over the sodium sulfate. The results of laboratory tests on the reproduction of embrittlement by means of caustic solutions are given. These show that embrittlement will not proceed in the absence of a stress above the yield point. Increased temperature causes the cracking to proceed at a faster rate in much more dilute solutions than heretofore used. Various steels have been tested to find if any one is resistant to this attack, and no steel has been found that is resistant to any marked degree. Heat treatment of the boiler plate does not stop the cracking. The inhibiting effect of sodium sulfate has been studied at a pressure of 500 lb. A theory covering embrittling action is also presented.

In recent publications[4, 5, 6, 7] there has been considerable discussion on the subject of embrittlement of boiler plate. This problem has been of much importance to the boiler manufacturer, the boiler user, and recently the water treating companies. Its occurrence is of such a nature that the detection of this trouble is hardly possible until the boiler is damaged beyond repair. The recent trend of the steam engineering field to higher steam pressures with the subsequent call for boilers free from scale has brought about the softening of waters which were heretofore not softened. Apparently in step with this increase in pressure and water treatment has come an increase in the number of cases of embrittlement, and with this increase, a demand for an explanation of the cause of the difficulty and methods to be used to prevent its occurrence.

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[1] Published by permission of the Director, Engineering Experiment Station, University of Illinois, Urbana, Ill.

[2] Professor of Applied Chemistry (Retired), University of Illinois, Urbana, Ill.

[3] Special Research Assistant, Engineering Experiment Station, University of Illinois, Urbana, Ill.

[4] *Bulletin 155*, Engineering Experiment Station, University of Illinois.

[5] *Kruppsche Monatsheffte*, Vol. 7 (November, 1926).

[6] *Mechanical Engineering*, Mid-November, 1926, pp. 1368–1372.

[7] *Speisewasserflege*, published by Union of Large Boiler Owners, Berlin, Charlottenburg (1926).

The present investigation being conducted at the University of Illinois is the result of cooperation between a group of public utility interests in the Middle West and the Engineering Experiment Station. The preliminary results of this investigation were embodied in publications released last year.[1, 2]

Review of Field for 1926

New Embrittlement Cases:

During the year 1926, eight specific instances of embrittlement were brought to the attention of the authors and a complete personal investigation was made in six of them. All cases were attributed to the treatment of the feed water. It brought about an excess of sodium hydroxide in the boiler coupled with a low sodium sulfate content. In two cases the use of an excess of soda ash was stopped. In three cases the installation of acid treatment was started in order to maintain the required sodium sulfate to sodium carbonate ratio. Two installations have changed the entire system of treatment so as to reduce the caustic alkalinity. In the last case a thorough investigation is now under consideration to find out the best method to be used in preventing any recurrence of this trouble.

No trace of difficulty has been encountered in any of the plants which have taken precautions to maintain the recommended sodium sulfate to sodium carbonate ratio. The ratio which the A.S.M.E. Boiler Construction Code for 1926 has recommended is as follows:

> In view of the particular cases of embrittlement cited. . . . and pending further research, the maintenance of not less than the following ratios of sodium sulfate to the soda (methyl orange) alkalinity is recommended as a precautionary measure:

Working Pressure of Boiler, lb. gage	Relation of Sodium Carbonate Alkalinity to Sodium Sulfate
0–150	1 to 1
150–250	1 to 2
250 and over	1 to 3

Opinions of Foreign Investigators:

W. Rosenhain has been quoted as follows:

> "I do not know of any recent publication in this country bearing on the matter, but I have followed with interest what has been done in America and Germany. . . . The only light I can throw on the whole question is that, so

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[1] S. W. Parr and F. G. Straub, "The Cause and Prevention of Embrittlement of Boiler Plate," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 52 (1926).

[2] *Bulletin 155*, Engineering Experiment Station, University of Illinois (1926).

far, our experiments—made with a view of producing fracture under prolonged loading in mild steel stressed to various intensities at a temperature of 300° C. in air—have failed to produce fracture even after a number of years' exposure. This experiment certainly suggests that some subsidiary agency of a corrosive nature is necessary to produce failure in a reasonable time, although it is quite possible that the particular sample of steel which I employed was not one subject to intercrystallin fracture under prolonged loading."

Stromeyer,[1] in summing up the cases of boiler failures which have come to his attention, shows that a large number of cases of embrittlement which were attributed to other causes than caustic in the water can be and are now readily seen to be due to caustic in the boiler.

A large mass of information pertaining to steel becoming brittle under certain strain conditions as well as the intercrystallin cracking of boiler plate has recently been presented before meetings of the Union of Large Boiler Owners in Germany.

Bauman, Ries, and Thiel[2] all describe cases of intercrystallin cracking produced in apparatus having caustic solutions in contact with strained steel. The latter has shown that caustic soda can concentrate in the clearances between the boiler plates to a high degree with resulting reduction of tensile strength. Bauman[3] has stated that the presence of caustic is not essential to the formation of intercrystallin cracks, though he admits caustic and strain produce this type of cracking.

Fry[4] discusses a new type of steel which does not become brittle after cold working, and ascribes embrittlement to the aging of the boiler plate. He states that aging not only produces embrittlement but also causes the service cracks developed in the presence of boiler feed waters. This shows that the term embrittlement is used to describe more than one type of cracking.

PRESENT STATUS OF OPINIONS ON EMBRITTLEMENT

It is agreed by all that there is a particular type of boiler distress which is characterized by intercrystallin failure in the riveted areas. Neither is it questioned that similar cracking is encountered in the strained areas of steel containers brought in contact with caustic. A slight complication of the issue is brought about by the use of the term "embrittlement" to mean the loss of ductility, irrespective of the type of cracking, and the producing of intercrystallin cracking in the absence of caustic. The general contention of the present investigation has been that strain and chemical action produce this type of cracking in a normal plate. The chemical action in boilers is brought about by the presence of sodium hydroxide, since no boiler operating with low sodium hydroxide and high sulfate has ever encountered this difficulty. The efforts of the authors have been directed toward the explanation of this cracking in boilers alone, and not trying to explain all cases of intercrystallin cracking. With this idea foremost it has been thought advisable to continue the use of the term "embrittlement of boiler plate."

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[1] The Manchester Steam Users' Association, Memorandum by the Chief Engineer for 1925.

[2] *Speisewassernege*, published by Union of Large Boiler Owners, Berlin, Charlottenburg (1926).

[3] R. Bauman, in a paper presented before the General Union of German Steam Boiler Inspection Companies, Zurich (1926).

[4] Fry, *Kruppsche Monatsheffle*, Vol. 7, November, 1926.

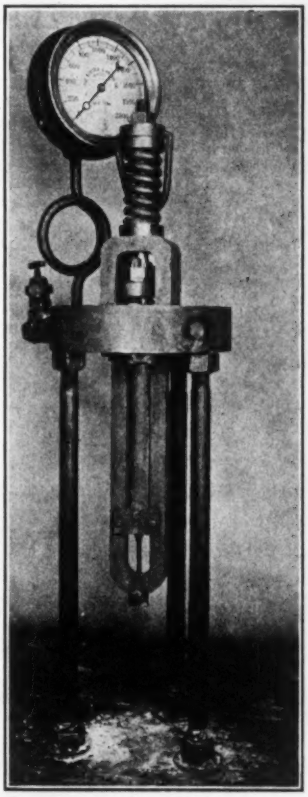

FIG. 1.—Top Section of Testing Unit.

For the purpose of confining the issue to the definite problem at hand, "embrittlement" as discussed here is limited to that condition which is characterized by the intercrystallin cracking of normal boiler plate in the riveted areas. It is realized that brittleness of boiler metal can be brought about by various means and the same metal can be made to crack under various methods of treatment; but the final test when comparison is made with this type of boiler distress should be, are the cracks intercrystallin or not?

LABORATORY RESULTS

The results previously published showed that for steam pressures up to 150 lb., normal boiler plate would crack when stressed above the yield point in the presence of a sodium hydroxide solution of 350 g. per liter or approximately 20,000 grains per gal. Solutions of other salts encountered in the boiler had no effect on the steel under similar conditions. The presence of sufficient sodium sulfate with the sodium hydroxide stopped the cracking.

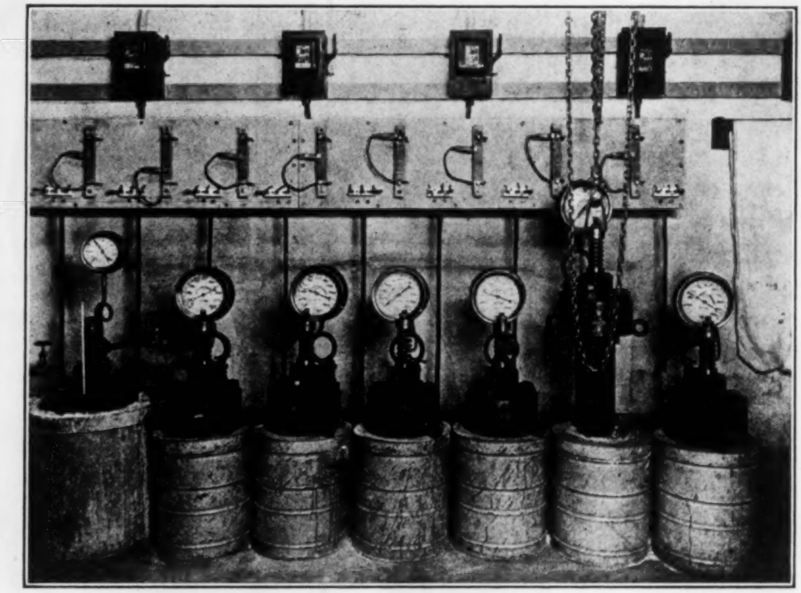

FIG. 2.—Equipment Used in the Experiments on Reproduction of Embrittlement.

In order to obtain results which might be correlated with operating conditions of boilers using higher steam pressures, apparatus was designed which would allow tests to be conducted at pressures up to 1500 lb.

Figure 1 shows the interior construction of the new test containers. The general idea of spring load used in the previous tests was continued. The only change was in the design of the container so as to withstand the higher pressures. Figure 2 shows some of the test units with electric heating furnaces and control panel. These new containers were furnished through the courtesy of the Babcock and Wilcox Co.

The size and shape of the test specimens were kept the same as in the previous tests.

TABLE I.—CHEMICAL ANALYSES OF METALS TESTED.

Reference	Material	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Nickel, per cent
F. S.	Flange Steel	0.18	0.45	0.012	0.027	.....	....
M. I.	Magnetic Iron	0.023	0.017	0.003	0.010	0.01	....
Ni	Nickel Steel	0.18	0.54	0.035	0.028	0.23	2.62
Si	Silicon Steel	0.34	0.75	0.027	0.034	0.29	....
Mn	Manganese Steel	0.35	2.13	0.020	0.026	0.023	....
Acid	Acid Open-Hearth Steel	0.42	0.46	0.036	0.044	0.051	....
W	From Embrittled Boiler Plate	0.22	0.55	0.015	0.035	.....	....
G	German Plate	0.06	0.33	0.012	0.035	.....	....
E	English Steel	0.16	0.35	0.018	0.018	0.040	....
1020	S.A.E. Steel No. 1020	0.18	0.56	0.012	0.040	0.005	....
1112	S.A.E. Steel No. 1112	0.15	0.65	0.086	0.156	0.028	....

TABLE II.—TENSION TESTS OF MATERIALS USED.

Reference	Description	Heat Treatment	Yield Point, lb. per sq. in.	Tensile Strength, lb. per sq. in.
F. S.	Flange Steel	As received	35 200	60 400
M. I.	Magnetic Iron	Annealed at 950° C	30 000	50 700
Ni	Nickel Steel	As received	45 000	75 300
Si	Silicon Steel	As received	43 000	87 000
Mn	Manganese Steel	As received	47 000	103 000
Acid	Acid Open-Hearth Steel	As received	33 000	74 000
W	From Embrittled Boiler Plate	As received	33 400	65 400
G	German Plate	As received	32 000	56 500
E	English Steel	Annealed at 950° C	27 000	50 000
1020	S.A.E. Steel No. 1020	Annealed at 950° C.	31 000	56 000
1112	S.A.E. Steel No. 1112	Annealed at 950° C	41 000	63 500
Sorbitic	Flange Steel	Oil quenched from 850° C.; reheated to 600° C	53 000	68 600
Spheroidized	Flange Steel	Oil quenched from 850° C.; reheated to 675° C. for 8 hours	50 600	61 100

Tests Conducted:

Series of tests were conducted toward answering the following questions in regard to the embrittling action:

1. Can a time factor be established which will serve to indicate the possibility of cracking at lower concentrations over prolonged periods?

2. Is the rate of cracking dependent on the stress; and is it possible to predict whether cracking will take place over a long period of time at a stress below the yield point?

3. What is the effect of changing the chemical composition of the steel?

4. Can a retarding influence be produced by heat treatment of boiler plate?

5. What influence does increased steam pressure have upon the rate of cracking?

6. What influence does the addition of sodium chloride have upon the time of cracking?

The tests that were conducted to answer these questions were run with special precautions being taken to guard against results which might not be representative. The total number of specimens tested amounts to around 300. Duplicate tests were made wherever the results were such that definite conclusions were to be drawn. Specimens which did not crack were later cracked with standard solutions and stresses which showed that they were not resistant to the general attack.

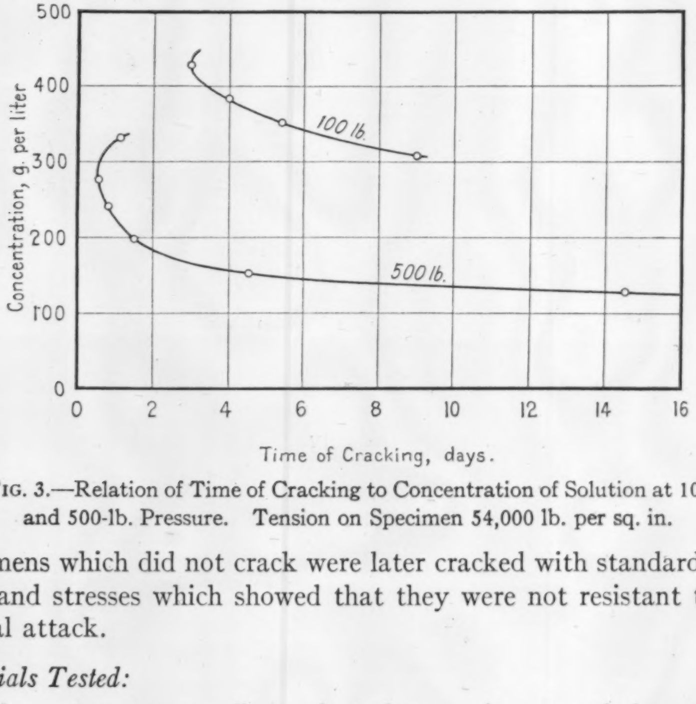

FIG. 3.—Relation of Time of Cracking to Concentration of Solution at 100 and 500-lb. Pressure. Tension on Specimen 54,000 lb. per sq. in.

Materials Tested:

The tests were normally conducted on specimens made from standard boiler plate. Special tests were run on steels of various chemical composition. The chemical composition and reference numbers of the various steels tested are given in Table I; Table II gives the physical properties.

DATA FROM TESTS

Relation of Time of Cracking to Concentration of Solution:

Tests were run at 100 and 500-lb. steam pressure. The results of these tests are shown in the graph in Fig. 3. These results indicate that the time of cracking increases with decrease of concentration and decreases with increase in temperature. The curve at 500-lb. pressure shows that over a prolonged period it is not unreasonable to assume that cracking could take place at a concentration as low as 80 g. per liter, approximately 5000 grains per gal. This concentration is only twenty times greater than that encountered in the body of the boiler.

TABLE III.—EFFECT OF TOTAL LOAD ON TIME RATE OF CRACKING.

PRESSURE 500 LB., SOLUTION 300 G. NaOH PER LITER.

Total Load, lb.	Time of Cracking	Time Rate of Cracking, sq. in. per hr.
54 000	12 hours	0.0004
54 000	12 hours	0.0004
44 000	24 hours	0.0005
35 500	36 hours	0.0005
33 000	No crack at 35 days	0.0000

TABLE IV.—EFFECT OF CHEMICAL COMPOSITION OF STEEL UPON TIME RATE OF CRACKING.

PRESSURE 500 LB., SOLUTION 300 G. NaOH PER LITER.

Steel Tested	Yield Point, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Total Load During Test, lb.	Time of Cracking	Time Rate of Cracking, sq. in. per hr.
F. S.	35 200	60 400	54 000	12 hours	0.0004
F. S.	35 200	60 400	35 500	36 hours	0.0005
M. I.	30 000	50 700	45 000	7 hours	0.0005
Ni	45 000	75 300	57 000	22 hours	0.0005
Si	43 000	87 000	60 000	8 hours	0.0018
Mn	47 000	103 000	64 000	22 hours	0.0008
Acid	33 000	74 000	54 000	12 hours	0.0010
W	33 400	65 400	54 000	24 hours	0.0004
G	32 000	56 500	40 000	13 hours	0.0010
E[a]	.....	.....	.....	.....	.....
1020	31 000	56 000	49 000	10 hours	0.0004
1112	41 000	63 500	54 000	2¼ days	0.0001

[a] Specimen too small for regular tests.

Effect of Total Stress on Time Rate of Cracking:

If it is assumed that the final rupture is purely a static failure produced when the intercrystallin cracking has weakened the metal until stress produced by the spring is sufficient to cause failure, then the rate of penetration of the intercrystallin cracking can be calculated.

In order to determine this rate the following calculations were used:

$$T = \frac{a - \frac{L}{u}}{t}$$

where T = time rate of cracking in square inches per hour;

a = area of specimen in square inches;

L = total load on plunger in pounds (spring plus steam);
u = ultimate tensile strength of specimen in pounds per square inch;
t = time of cracking in hours.

The results of tests run at 500-lb. pressure and at the optimum concentration, 300 g. per liter, with varying intensities of stress are tabulated in Table III. These results indicate that for the steels tested the time rate of cracking is independent of the total stress once the stress passes the region of the yield point. Below the yield point no cracking occurs. This shows that apparently no cracking can be predicted even over long periods of time when the localized stress is below the region of the yield point of the boiler plate.

TABLE V.—EFFECT OF PREVIOUS HEAT TREATMENT.

PRESSURE 500 LB., SOLUTION 300 G. NaOH PER LITER.

Steel Tested	Yield Point, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Total Load During Test, lb.	Time of Cracking	Time Rate of Cracking, sq. in. per hr.
F. S.	35 200	60 400	54 000	12 hours	0.0004
Spheroidized	50 600	61 100	54 000	12 hours	0.0004
Sorbitic	53 000	68 600	59 000	14 hours	0.0005
Sorbitic	53 000	68 600	49 000	28 hours	0.0005
Sorbitic	53 000	68 600	38 000	No crack at 20 days	0.0000

Effect of Composition of Steel:

Specimens of all the steels listed in Tables I and II were tested and their corresponding time rate of cracking determined. These figures are given in Table IV. The results show that all the steels tested crack within a reasonable time. The acid, German, silicon, and manganese steels crack at an appreciable faster rate than the normal boiler plate. The other steels, including that from an embrittled boiler, all had about the same time rate of cracking. Particular attention should be paid to steel No. 1112, a high phosphorus and sulfur steel, which had a time rate much lower than those of pure iron and boiler plate. This indicates that even the high sulfur and phosphorus content does not make the steel more susceptible to cracking of this nature.

Effect of Previous Heat Treatment:

The suggestion has been made that a heat treatment which will make the grain boundaries very rough or indistinct, such as exists in the sorbitic and spheroidized condition, might have a retarding influence on the cracking.[1] Tests were accordingly run on samples of flange steel previously heat treated to bring about these types of structures with the results as tabulated in Table V. The presence of the sorbitic or spheroid condition does not retard the cracking of the boiler plate. One point of particular interest is that making the steel sorbitic raises the yield point. The cracking still takes place below this point but above the yield point of the untreated metal. This shows that the cracking is not a real function of the yield point but happens to require an initial stress which is in the region of the yield point. The results obtained from testing cold-rolled specimens previously reported,[1] showed that raising the yield point by cold work

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[1] W. Rosenhain, private communication.

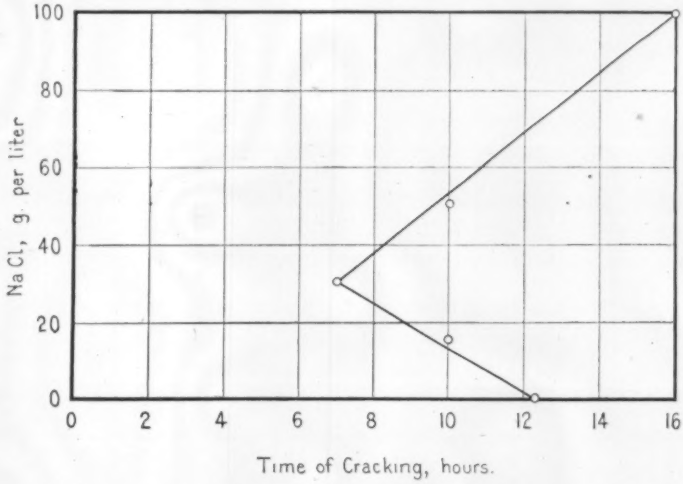

FIG. 4.—Relation of Time of Cracking to NaCl Added at 500-lb. Pressure. Sodium Hydroxide, 300 g. per liter. Tension on Specimen 54,000 lb. per sq. in.

also raised the stress necessary for embrittlement, but not in proportion to the rise in yield point produced by the cold work.

Effect of Higher Steam Pressures on Rate of Cracking:

The curves in Fig. 3 show that the rate of cracking is much faster at 500-lb. pressure than at 100-lb., and that another factor is introduced in that the concentration of solution is entirely different for the optimum cracking time. If the high concentrated solutions necessary for the lower pressures are used at the higher pressures the solutions attack the steel with evolution of hydrogen and necessitate the stopping of the test. When the lower concentrations are used the time rate at the lower pressures becomes too low for testing. In general, it can be stated, that at higher steam pressures the cracking takes place with a marked increase of rate even with a much more dilute solution of sodium hydroxide.

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[1] S. W. Parr and F. G. Straub, "The Cause and Prevention of Embrittlement of Boiler Plate," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 52 (1926).

Effect of Adding Sodium Chloride to the Hydroxide:

Tests conducted with varying amounts of sodium chloride added to the sodium hydroxide at 500-lb. pressure and 300 g. of sodium hydroxide per liter gave results which are shown in graph form in Fig. 4. This shows that sodium chloride added in small amounts accellerates the cracking under the conditions tested, while larger amounts tend to reduce this accellerating effect. When 150 g. of sodium chloride per liter was added a rapid generation of pressure occurred as soon as 500-lb. pressure was reached. The removal from the source of heat caused this pressure to drop back and when reheated it returned. Pure sodium chloride solution did likewise. No residual gas remained on cooling. This eliminated the possibility of hydrogen being generated.

TABLE VI.—EFFECT OF RATIO OF Na_2SO_4 TO NAOH AT 500-LB. PRESSURE.

Solution, g. NaOH per liter	NaCl, g. per liter	Ratio, Na_2SO_4 to NaOH	Stress, lb. per sq. in.	Time of Cracking	Ratio, Na_2SO_4 to alkalinity as Na_2CO_3
135	25	0	50 000	$1\frac{1}{2}$ days	0
120	24	1.6	50 000	$1\frac{3}{4}$ days	1.1
127	24	2.1	50 000	3 days	1.5
120	25	2.6	50 000	3 days	1.8
140	27	3.1	50 000	2 days	2.2
130	24	4.0	50 000	4 days	2.8
135	25	5.0	50 000	No crack at 30 days	3.5

LABORATORY RESULTS ON INHIBITION OF CRACKING

The previous work on the inhibiting effect showed that to produce this effect the solution must be brought around the specimen in such a manner that any salt precipitating out will form on the test specimen. To do this the specimen was entirely surrounded by steel, allowing but $\frac{1}{32}$ in. of space between the specimen and the surrounding steel. A dilute solution of the salts was started with and this slowly concentrated by the removal of steam. When the proper caustic concentration was reached the test was started.

To adapt this to the higher pressure, tests were conducted using the lowest caustic concentration which would produce cracking. This was done so that if it is a solubility effect, the lower the sodium hydroxide content the higher will be the solubility of the sulfate. In this manner the most protecting ratio can be obtained. If sodium chloride is also introduced then the best conditions for cracking are obtained, and if the sulfate can stop this ideal condition, the conclusion can be drawn that the ratios obtained are protective.

When a solution of 125 g. of sodium hydroxide per liter and 25 g. of sodium chloride per liter was used the time of cracking was about 1¾ days at 500-lb. pressure. The introduction of the NaCl had decreased the cracking time from 14½ days to this short period. This solution was adopted as the standard to be maintained for the sulfate tests at 500-lb. pressure. The solution to be used was made up dilute and then concentrated under pressure by removing steam. The results obtained are given in Table VI. These results show that with a ratio of sodium sulfate to hydroxide up to 4.0 or to alkalinity as carbonate of 2.8 little inhibiting effect is produced at 500 lb. When this ratio is passed the inhibiting action is noticed.

The question of the inhibiting effect of the sulfate would seem to be that of solubility in the hydroxide. At lower pressures where higher concentration of hydroxide prevails the solubility of the sulfate is lower and less is required to inhibit the cracking. At higher pressures with lower concentration of caustic and higher temperatures the solubility of the sulfate is higher, thus requiring more to be present before precipitation takes place.

The solubility effect, however, may not be the ultimate dominating factor, and electrolytic potential may have equal or greater importance. In the latter case other inhibiting conditions may be found effective, and active study of this phase of the question is being prosecuted.

MECHANISM OF EMBRITTLEMENT

The results obtained are in accord with the ideas as set forth in the publication of the authors' previous results.[1] The steel has a definite electromotive force in respect to the sodium hydroxide. If this e.m.f. is sufficient the iron is attacked with the evolution of hydrogen and the formation of a coating of Fe_3O_4, magnetic oxide, on the steel. The e.m.f. of the steel coated with this oxide is low in respect to the caustic solution, but the fresh metal under this coat has a high e.m.f. towards the solution, which favors penetration. The steel under strain has the grain boundaries under high stress which adds to the already more chemically active property and accelerates penetration at these points.

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[1] S. W. Parr and F. G. Straub, "The Cause and Prevention of Embrittlement of Boiler Plate," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 52 (1926).

That the cracking is the result of an electromotive force between the steel and the solution is borne out by the results of various tests conducted. First, an increase in temperature causes an increase in the e.m.f. of the metal to the solution and consequently lowers the concentration necessary. Second, any salt which will destroy this e.m.f., such as chromate, when introduced, stops the cracking. Third, if the concentration is raised, hydrogen is generated freely and the cracking is stopped since the action is no longer selective due to too high an e.m.f. which favors general attack. Fourth, the time rate can be varied by using a container which has had the oxide coating removed, thus changing the electromotive force of the specimen in respect to the container.

From all the data available the following very brief and partially incomplete explanation is made at this time to account for this particular cracking.

The main essential is a solution which has an e.m.f. in respect to steel just sufficient to favor the reaction:

$$3\text{Fe} + 4\ \text{OH} = \text{Fe}_3\text{O}_4 + 4\ \text{H}$$

It must not be any higher than necessary to start this reaction at the temperature involved. When the metal is in an unstrained condition, a thin compact coat of oxide is formed which is slowly penetrated with the forming of a heavier coat and eventually the entire metal will be changed to the oxide. The attack is fairly even and penetrates the metal at an even rate at all points. If the metal is subjected to sufficient stress under these conditions the grain boundaries become active, first, from the increased chemical activity brought about through the energy stored up there by the stress, and second, by an increased e.m.f. produced at these points of high stress. If the electromotive force is just sufficient to favor the action on the metal, this slight increase at the grain boundaries becomes sufficient to favor a much more rapid penetration at this point. The products of the action Fe_3O_4 and H_2 both tend to favor the further penetration. As already pointed out by Williams and Homerberg,[1] the cathodic hydrogen will penetrate into the fine capillaries at the grain boundaries and reduce any oxides with the formation of water and an increase in volume thus increasing the stresses present at the boundaries. This product of chemical action does not plug the crevices and stop further chemical action but due to its e.m.f. relation to the solution and the fresh metal inside acts as a stimulating agent and increases the action at these points.

If the e.m.f. is high, such as produced by the action of an acid on a metal, the generation of H_2 is general and any slight difference in activity between the grains and the boundaries becomes negligible in the excess of e.m.f. the acid produced. When too concentrated a solution of caustic is used at high temperatures, the test specimen is corroded generally, and even when under strain does not crack. Thus, the attack has also been so general and vigorous that the influence of the small grain boundary effect is lost.

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[1] *Transactions*, Am. Soc. Steel Treating, April, 1924.

The retarding influence of sodium sulfate is easily explainable on this basis. The salt crystallizing out on the surface of the metal lowers the e.m.f. of the metal and stops the action. It is not the plugging effect, as some have suggested, thus keeping the solution away, but instead the salt plays the rôle of an insulator breaking the e.m.f. Any oxidizing salt like chromate will also retard action by destroying the e.m.f. If oxygen is present, the effect is also destroyed due to the fact that the oxygen tends to oxidize the ferrous salts in solution, which destroys the e.m.f. in respect to the steel.

CAUSE OF EMBRITTLEMENT IN STEAM BOILERS

The results of the experimental work show that the boiler plate tested when stressed below the region of the yield point will not crack when subjected to caustic attack. To think of stresses in boilers in the range of the yield point of boiler plate should not necessarily conflict with our ideas in regard to construction of boilers. It has been common knowledge for years that there are high concentrated stresses in the riveted areas of boilers. Thousands of boilers have operated with these stresses present until discarded. This is due to the fact that a ductile metal like boiler plate can endure localized stresses of this magnitude indefinitely. Naturally, it is well for the boiler manufacturer to reduce these stresses to as low a figure as possible in the future, but for the boilers already installed one can do little more than follow the operators' slogan, "keep operating."

The concentration curves show that cracking can take place at much lower concentrations than heretofore predicted, and over a period of long duration one might expect to encounter cracking if the caustic concentration reaches approximately 4000 grains per gal. This is but twenty times the actual concentration which may be reached in the boiler. Seams with small crevices between the plates, under the high temperature and pressure incident to boiler operation, and with small incipient leaks which act as reducing valves and allow the liquid to flash out as steam leaving the salts behind, must cause the solution to concentrate. Thiel has shown that these concentrations may reach as high as 75-per-cent NaOH in the seam of a boiler. Tests conducted in the authors' laboratory have shown the possibility of a solution of

200 grains of caustic per gal. being concentrated in a space between two cylinders to 7000 grains and still be in contact by means of a leak between the inside and outside with the original solution of 200 grains.

PREVENTION OF EMBRITTLEMENT

The actual ratios of salts for the prevention of embrittlement have not been determined to the point where minimum limits can be set. The results indicate that for normal pressures the A.S.M.E. recommendations are safe and should be adhered to.

TABLE VII.—INHIBITING EFFECT OF SODIUM PHOSPHATE ON EMBRITTLEMENT OF BOILER PLATE.

STRESS ON SPECIMEN, 45,000 LB. PER SQ. IN.; STEAM PRESSURE, 500 LB.

Solution, g. NaOH per liter	Sodium Phosphate ($Na_3PO_4 \cdot 10H_2O$), g. per liter	Time of Cracking	Length of Time Tested, No Cracking
295	0	24 hours	....
280	2	20 hours	....
280	5	....	17 days
280	0	20 hours	....
280	10	....	13 days
280	3	....	13 days
280	5	....	13 days
280	3	....	15 days

TABLE VIII.—INHIBITING EFFECT OF TANNATE ON EMBRITTLEMENT OF BOILER PLATE.

STRESS ON SPECIMEN, 45,000 LB. PER SQ. IN.; STEAM PRESSURE, 500 LB.

Solution, g. NaOH per liter	Tannate, g. per liter	Time of Cracking	Length of Time Tested, No Cracking
280	0	20 hours	....
270	50	....	10 days
275	5	20 hours	....
275	15	....	30 days
275	10	....	30 days

Methods of treatment for various types of waters and conditions of boiler operation are being investigated by the authors, having special reference to the development of further inhibitive conditions or reagents with the prospect of avoiding some of the present difficulties attending the maintenance of the sulfate ratio and also greatly simplifying the procedure for the prevention of this very serious menace.

Reference has already been made in this duscussion to the possibility of the importance of the electrolytic potential in controlling embrittlement. The use of sodium dichromate as an inhibitive agent was suggested in a bulletin of the Engineering Experiment Station of the University of Illinois[1] and its possible action attributed to its influence on the electro-potential of the steel to caustic solutions. With this idea foremost a list has been prepared of various chemicals which might be used in boiler waters to bring about this effect.

Tests have been conducted on a few of these salts and the results obtained have been very encouraging. The addition of sodium phosphate or tannate to sodium hydroxide lowers the electro-potential of steel to the solution. The addition of these salts to the regular caustic solutions used brought about the results tabulated in Tables VII and VIII.

The data presented in these tables are tabulated in the same order in which the tests were conducted. These results indicate that comparatively small amounts of these inhibitive salts will retard the embrittling action of sodium hydroxide. Thus, as low as 3 g. per liter of sodium phosphate ($Na_3PO_4 \cdot 10H_2O$) prevented embrittlement in the presence of approximately 300 g. per liter of sodium hydroxide at a steam pressure of 500 lb. When sodium sulfate was used at this pressure it was necessary to have five times as much sodium sulfate as sodium hydroxide. The phosphate appears to be effective as an inhibitant and requires only one part of sodium phosphate to 100 parts of sodium hydroxide—less than $\frac{1}{500}$ as much as the sodium sulfate. Similar figures can be shown for the use of tannate. Tests are being conducted with other salts as possible inhibiting agents.

The possibility of the use of these new inhibitants is at once evident, but their application in boiler feed water treatment should not be tried until more laboratory work is in evidence and more data can be collected from plant operation. It should be realized that these figures represent data collected from the accelerated laboratory tests and are not directly applicable to operating conditions.

The development of special boiler plate which would be resistant to the caustic attack is one method of stopping this difficulty. The authors have had this in mind and have made attempts to secure samples of various types of steels and see if any were resistant to this attack. The results of these tests have been given. Fry[2] discussed a new type of steel developed by the Krupp Works in Germany which resisted embrittlement when tested in contact with hot caustic solution. A sample of this steel was received about the first of June through the courtesy of Mr. A. G. Christie of Johns Hopkins University. The chemical analysis, as furnished by the Krupp Works, is as follows:

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[1] *Bulletin 94*, Engineering Experiment Station, University of Illinois, 1917, p. 46.

[2] *Kruppsche Monatsheffte*, Vol. 7 (November, 1926).

Carbon	0.15	per cent
Manganese	0.44	"
Phosphorus	0.017	"
Sulfur	0.023	"
Silicon	0.04	"
Nickel	0.026	"

The physical properties obtained were as follows:

Yield point	32 000	lb.	per sq. in.
Tensile strength	56 000	"	"

This steel when subjected to the regular embrittlement test cracked at a time rate of 0.0004 sq. in. per hour, a time rate identical with that obtained from testing the regular American plate. The older German plate cracked at a time rate of 0.0010, which is much faster. Consequently it can be said that the sample of steel obtained is no more resistant to embrittlement than the regular American boiler plate.

DISCUSSION

MR. D. J. MCADAM, JR.[1] (*presented in written form*).—This paper is of great practical value and is also of theoretical interest. The term "embrittlement," however, seems somewhat unfortunate. There is no evidence that true embrittlement (such as the brittleness of glass) is responsible for any of the phenomena described. It is true that under the influence of the caustic solutions there is loss of ductility, but this is due to loss of continuity of the metal, not to brittleness. This term is unfortunate also for another reason. It tends to give the impression that this type of failure of boiler plate differs radically from other types of failure under combined stress and chemical action. I believe it should be emphasized that this is merely one phase of a large subject, the influence of simultaneous stress and corrosion.

Mr. McAdam.

In support of this idea I shall refer briefly to a boiler failure last August at the Naval Engineering Experiment Station. This Mosher boiler had been used at irregular intervals for safety valve tests. It had been in service about eleven years and in that time had been used about 1000 times. Sometimes the intervals of idleness would be several months.

The boiler water used was a temporary hard water containing principally calcium bicarbonate, the same water that is being used in corrosion-fatigue tests. No boiler compound had ever been used in the boiler.

Last August a boiler head blew out at 350 lb. pressure, causing great damage, killing two men and injuring others. The boiler head failed at the "knuckle," the junction of the flange and head. In this region there was no seam. Examination showed that a crack had been progressing slowly (probably for years) from the inner to the outer surface. At the time of the explosion the crack in some regions had advanced to within $\frac{1}{32}$ in. of the outer surface. This crack was entirely below the water level. The head had been held on, probably for months or years, chiefly by the intact metal above the water level.

Examination of the other head of the boiler showed similar shorter cracks almost continuous around the lower half of the "knuckle." All were below the water level. The action of the water, therefore, was one factor involved in the failure of this boiler.

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[1] Metallurgist, U. S. Naval Engineering Experiment Station, Annapolis, Md.

Mr. McAdam. The other factor was the relatively high stress at the knuckle. The plate was thinner than it should have been. At full pressure the stress at the knuckle may have been 20,000 or 25,000 lb. per sq. in. As previously stated the stress had been applied and removed about 1000 times in about eleven years.

The failure of this boiler, therefore, was evidently due to combined corrosion and repeated stress at elevated temperature. This failure, occurring during the investigation of corrosion-fatigue, naturally has been viewed as an example of corrosion-fatigue. In this boiler the stress cycles were of very low frequency, so that the amount of corrosion per cycle was great. Under such conditions fewer cycles were needed to cause failure than if the cycle of frequency had been greater. The corrosion-fatigue experiments at the Naval Experiment Station have been at cycle frequencies from 4 to 1450 per minute, with complete reversal of stress per cycle, and at room temperature. In boilers, the stress range is from zero to a maximum and at elevated temperatures. Experiments are greatly needed to determine the corrosion-fatigue limit of steel under such conditions.

It appears to me that the factors involved in the failure of boilers containing caustic soda differ chiefly in degree from the factors involved in the boiler failure that I have just described. In boilers containing caustic soda the intensity of chemical attack is probably greater than in the boiler that failed at Annapolis. Otherwise the process is essentially similar.

I believe that it is possible to lay too much emphasis on the intercrystallin nature of some failures. Evidence indicates that whether a corrosion-fatigue failure is intercrystallin or not depends on several factors such as cycle frequency, chemical composition, etc. It seems entirely possible that corrosion-fatigue failure of boilers may sometimes be intercrystallin but not always so.

It would be of interest if the authors would continue their experiments using stress cycles from zero to a maximum. Possibly under such conditions failure would occur at lower maximum stress than if the stress is steady.

It is of interest to note that the authors find that the steady stress necessary to cause failure is little if any changed by heat treatment. This is analogous to the results of corrosion-fatigue tests of steel. The corrosion-fatigue limit of carbon steels is little affected by heat treatment. Nothing is known about the relation between the steady stress necessary to cause corrosion cracks and the corrosion-fatigue limit. This subject should be investigated.

These remarks are not intended to minimize the value of this very important paper. They merely express a slightly different viewpoint on some phases of the subject. **Mr. McAdam.**

I think the authors are on the right track in considering the subject from the point of view of the electrolytic potential. Electrolytic potential seems to be an important factor in corrosion-fatigue.

Mr. Speller. MR. F. N. SPELLER.[1]—It is to be regretted that the papers on corrosion-fatigue and caustic embrittlement, so-called, were not presented and discussed at the same time. Last year attention was called to the possibility of these being different phases of the same phenomena, namely, chemical attack combined with variable or static stresses. Since then we have done more work on the effect of corrosion inhibitors in general. For instance, disodium-phosphate has been found very effective in inhibiting the action of brine and other solutions at normal temperatures. All these subjects seem to have a certain relation, and should be discussed together.

We are now at work on a study of inhibitors that can be used in strong acids at normal temperatures, and while nothing is known positively as to the cause of the protective action of these inhibitors, the results have already found quite a useful field of application. As a practical illustration, we have applied this principle, using 15-per-cent hydrochloric acid with an inhibitor at 120° F. to clean the entire water-pipe system of a skyscraper in New York City, without any noticeable action on the steel pipe.

Mr. Hall. MR. J. H. HALL.[2]—My discussion applies merely to one of the terms used in this paper. Since 1892 the company which I represent has been making what we call manganese steel, which contains 12 per cent of manganese. Ten years ago a steel containing 2.35 or 2.5 per cent manganese was not known as manganese steel, in fact, it was not made. Now, I see mention in this paper of a steel called manganese steel which contains 2.35 per cent of manganese.

The United States Government to-day is specifying manganese steel when they mean a steel containing 1.5 to 2 per cent manganese. I had a discussion about this with Mr. Gilligan of the Henry Souther Engineering Co. a year or two ago, with a view to arriving at some term to describe these lower manganese steels. If 12-per-cent metal is manganese steel, surely this 2.35-per-cent metal is not.

It seems to me that some committee on nomenclature should look into the question of finding names for these several grades of steel, and it has occurred to me that possibly our Society would be willing to undertake the task.

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[1] Metallurgical Engineer, National Tube Co., Pittsburgh, Pa.

[2] Metallurgical Engineer, Taylor-Wharton Iron and Steel Co., High Bridge, N. J.

Mr. Parr. MR. S. W. PARR.[1]—It might be suggested to include also a definition as to what is nickel steel.

Mr. Cone. MR. E. F. CONE.[2]—Along that line the annual statistics of the American Iron and Steel Institute on rails recently reported something like fifteen thousand tons of manganese rails made in 1926, and that was immediately interpreted by some of us to mean 1.5-per-cent or medium manganese steel. Since then it has developed that it was wrongly reported as manganese steel, although it was that class of material, and hereafter the institute will report as manganese steel only material containing over 10 per cent. Are not steel rails containing 1.5 or 1.25 per cent, or whatever it is, really manganese steel of a different kind and should this steel not be classified as alloy steel? It seems to me that the Society could take up that question and define what manganese steel is. We have to-day two kinds of manganese steel, the 1.25 to 1.75 per cent or "medium" and the higher manganese (12 to 14 per cent).

Mr. Flowers. MR. A. E. FLOWERS.[3]—Regarding the question of nomenclature raised by Mr. Hall, may it not be possible to use a term already somewhat in use, and say manganese-bearing steels, for those containing about one to two per cent of manganese, and keep the term which we now all understand as manganese steel, for those containing about 12 per cent manganese?

Mr. Kenney. THE CHAIRMAN (*Mr. E. F. Kenney*).[4]—I think as a matter of interest, I might say that the railroads have tried to take care of this by calling those steels approximating 1.5 per cent in manganese, medium-manganese steels, and rails made of that grade of steel are stamped with initials which indicate that they are medium-manganese steel.

Mr. Straub. MR. F. G. STRAUB[5] (*author's closure by letter.*)—The type of failure described by Mr. McAdam has received considerable attention by the A.S.M.E. Boiler Code Committee. At present the boiler manufacturers do not make the boiler heads with such a sharp bend at the knuckle, and I believe they have entirely eliminated this difficulty in their newly designed heads. In the past we have investigated cases similar to this one and have failed to find any tie up between them and embrittlement. In our paper last year attention was called to this type of failure where corrosion is accelerated by stresses of high magnitude and we showed why they were not to be classed with embrittlement cases.

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[1] Professor of Applied Chemistry (Retired), University of Illinois, Urbana, Ill.

[2] Associate Editor, *The Iron Age*, New York City.

[3] Engineer, In Charge of Development, The De Laval Separator Co., Poughkeepsie, N. Y.

[4] Metallurgical Engineer, Bethlehem Steel Co., Inc., Bethlehem, Pa.

[5] Special Research Assistant, Engineering Experiment Station, University of Illinois, Urbana, Ill.

SOME TESTS OF A CHROME-NICKEL STEEL AT HIGH TEMPERATURES

BY R. S. MACPHERRAN[1]

The tests here shown are a continuation of our work on testing steels at high temperatures, a description of which was given[2] before the Society in 1921. The heating box is about the same as shown in that paper, except for the small hole in the middle of the box through which a steel spring held the thermocouple against the test specimen. In our present box this hole has been omitted. The thermocouple wires now lead down from the top of the box and are in contact with the center of the test specimen. All specimens are held at constant temperature for about twenty minutes before test.

The purpose of these tests was to determine the forging and upsetting properties of a certain high-chrome high-nickel steel This steel was a $\frac{7}{8}$-in. round bar forged from $5\frac{1}{4}$-in. square and annealed at 1625° F. (885° C.).

The chemical composition, and the physical properties at room temperature were:

Carbon, per cent	0.46
Manganese, per cent	0.58
Silicon, per cent	0.91
Chromium, per cent	7.19
Nickel, per cent	18.73
Tensile strength, lb. per sq. in.	105,550
Elongation in 2 in., per cent	33
Reduction of Area, per cent	53

Our first series of tests were made using a base metal couple, but the results showed such a decided change at slightly over 1800° F. (980° C.) that the field was again covered, this time by a platinum couple so fastened that the junction was in actual contact with the center of the test specimen. It will be noted that at over 1400° F. (760° C), this material not only falls off in strength, but becomes less ductile until a temperature of about 1800° F. (980° C.) is reached. From this point the tensile strength continues to fall off, but the ductility increases to a remarkable extent. The time element is also a factor. A specimen held at 1800° F. (980° C.) for nearly three hours showed about the ductility of a specimen held for 20 minutes at 1850° F. (1010° C.).

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[1] Chief Chemist, Allis-Chalmers Manufacturing Co., West Allis, Wis.

[2] R. S. MacPherran, "Comparative Tests of Steels at High Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 852 (1921).

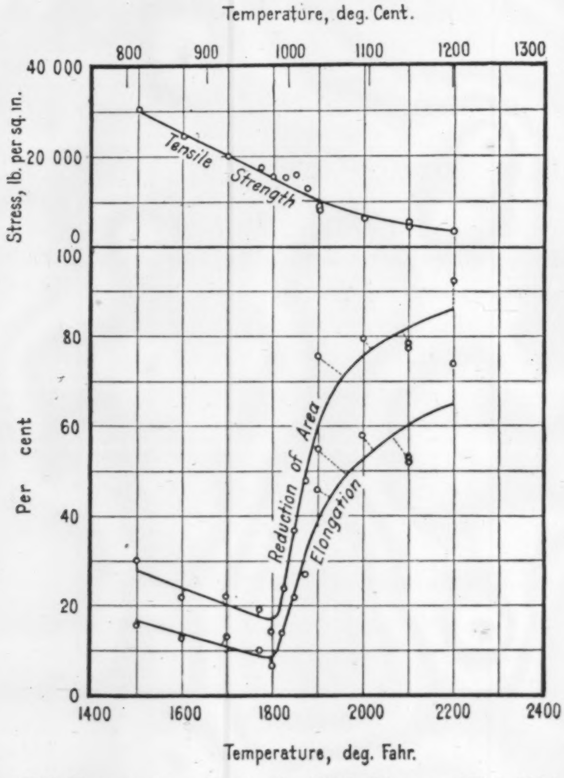

Fig. 1.—High Temperature Tests of a High-Chrome, High-Nickel Steel.

For straight line forging it was found that 2200° F. (1205°C.) was satisfactory, but for upsetting it was necessary to go to 2500° F. (1370° C.).

These tests are given in the hope they may be of interest to others who might wish to determine the physical properties of various alloy steels at or near the forging range.

DISCUSSION

Mr. Mochel.

Mr. N. L. Mochel[1] (*presented in written form*).—The results of Mr. MacPherran's tests are of great interest and of value if one is hot working steel of the same or similar composition.

He refers to his paper presented in 1921 in which results were given of tests at lower temperatures. Figure 5(*b*) covers the tests on presumably the same steel as is dealt with in the present paper, and gave results for tensile strength, elongation and reduction of area.

Tests were recently made at the Westinghouse Research Laboratory by Mr. McVetty on steels of approximately the same composition as given by Mr. MacPherran. The same methods were used as have been reported elsewhere [2, 3]. As the tests included the determination of proportional limit, Johnson elastic limit, yield point and elastic modulus, it is felt the results may be of interest.

The accompanying Fig. 1 shows the results of tests on material M-97 of the following composition:

Carbon	0.44	per cent
Manganese	0.63	"
Phosphorus	0.022	"
Sulfur	0.025	"
Silicon	0.87	"
Nickel	19.67	"
Chromium	7.88	"
Copper	0.038	"

The accompanying Fig. 2 shows the results of tests on material M-98 of the following composition:

Carbon	0.465	per cent
Manganese	0.76	"
Phosphorus	0.030	"
Sulfur	0.016	"
Silicon	1.67	"
Nickel	21.86	"
Chromium	7.83	"
Copper	0.76	"

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[1] Metallurgical Engineer, Westinghouse Electric and Manufacturing Co., Philadelphia, Pa.

[2] T. D. Lynch, N. L. Mochel, P. G. McVetty, "The Tensile Properties of Metals at High Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 5 (1925).

[3] P. G. McVetty and N. L. Mochel, "The Tensile Properties of Stainless Iron and Other Alloys at Elevated Temperatures," *Transactions*, Am. Soc. Steel Treating (1927).

Mr. Mochel. In Fig. 1 a method of plotting has been adopted to show the variation in results from several specimens, rather than attempting to draw a single line. It will be observed in Fig. 2 that the curves of elongation and reduction of area show a rapid decrease of ductility when the temperature exceeds 750° F. (399° C.)

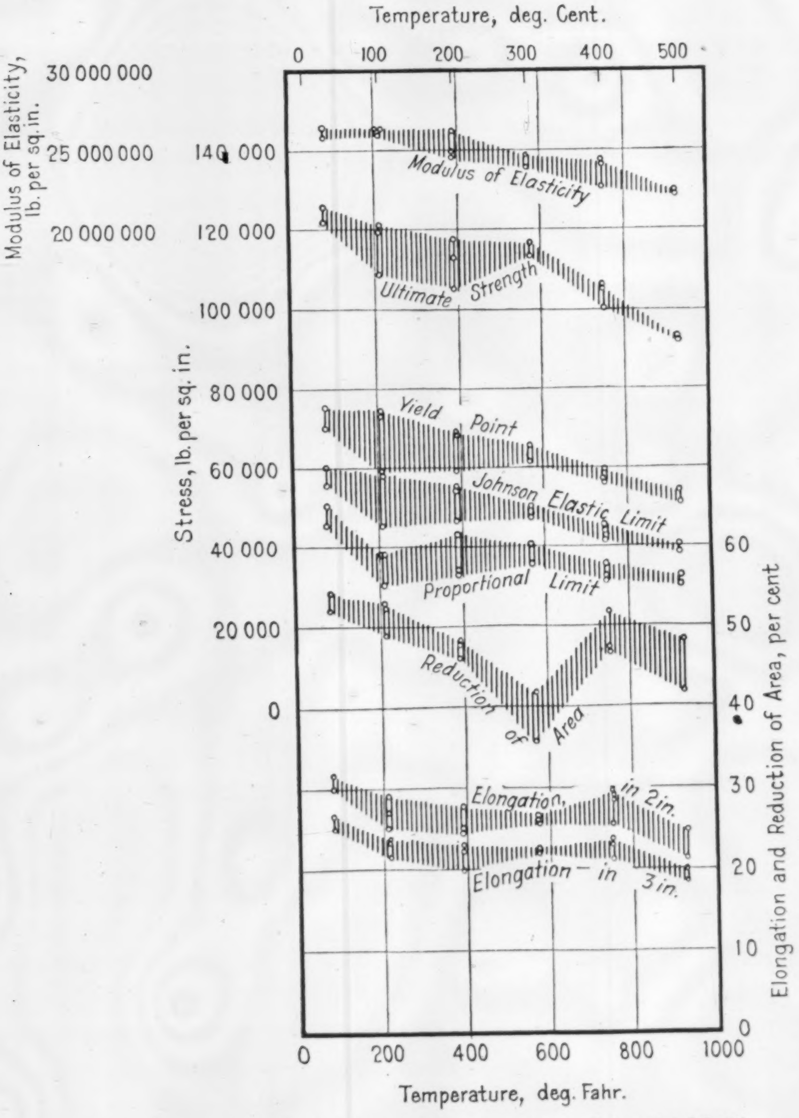

FIG. 1.—Tension Tests on Nickel-Chromium Steel (Material M-97).

Mr. Mochel. Relative to Mr. MacPherran's selection of temperatures for forging and upsetting, it would appear to the writer that the upsetting temperature is far too high. The temperature would depend on the character and amount of work to be done, but for any purpose it is believed 2500° F. (1371° C.) is too high and would be harmful to the material. It has been found by the writer that exhaust valves can be upset from similar material from a temperature of not over 2100° F. (1149° C.).

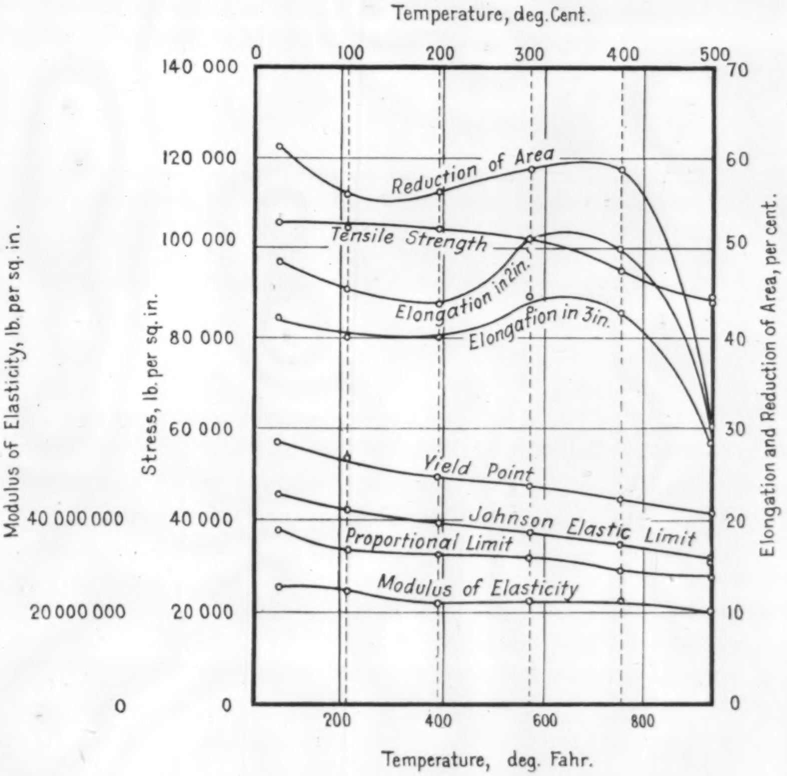

FIG. 2.—Tension Tests on Nickel-Chromium Steel (Material M-98).

Mr. MacPherran. MR. R. S. MACPHERRAN[1] (*author's closure by letter*).—The discussion by Mr. Mochel is very interesting. The physical tests he reports, however, were made at a much lower temperature than those in the paper just presented. His range of temperature is from 70 to 932° F. (21 to 500° C.). Our range is from 1400 to 2200° F. (760 to 1205° C.). The upsetting referred to was a very difficult job. For ordinary upsetting we do not exceed 2200° F. (1204° C.).

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[1] Chief Chemist, Allis-Chalmers Manufacturing Co., West Allis, Wis.

HIGH-TEST ALLOY CAST IRON

By M. E. Greenhow[1]

Synopsis

The term high-test alloy cast iron as described in this paper is the result of considerable research work carried on in 1923 in an effort to produce an iron by the cupola melting process, that would have the following properties and characteristics: Low total carbon and even distribution of graphitic carbon which is conducive to uniformity of structure and correspondingly high tensile strength. An iron of this description has the following commercial advantages: Easy machineability with a high Brinell hardness number, a minimum amount of grain growth and high resistance to wear, under proper lubrication, in equipment such as Diesel and gas engine cylinders, air compressor and brake cylinders, heads, liners, and other parts subjected to wear and high stress at elevated temperatures.

General Description

In 1923, considerable difficulty was encountered in obtaining in certain types of castings, varying in section from ¼ in. to 1 in., a close-grained, high-strength, wear-resisting cast iron, free from internal shrinkage. The silicon required to keep the thin section from chilling gave an undesirable structure, so considerable experimental work was carried on with various alloy additions until an iron with the desired physical properties was developed. The following is a brief summary of the physical characteristics and melting operation used.

The best results are obtained when the total carbon is between 2.75 and 3.00 per cent. It is not difficult to obtain this range of carbon with a large percentage of steel scrap, well balanced cupola construction, and efficient and uniform cupola operation. The manganese content should be from 0.75 to 0.90 per cent, its chief function being to eliminate oxides in the molten metal. The manganese lost in melting seems to be somewhat higher than in ordinary cast iron mixtures, which the author has found to be consistently 18 to 20 per cent of the total amount charged. while in the high-strength cast iron practice the percentage of manganese lost is from 25 to 29 per cent.

A fairly wide range of silicon content is permissible in this iron without any appreciable effect upon the strength, the most uniform results being obtained with a silicon content of 1.25 to 1.65 per cent. The author's experience is that a silicon content lower or higher than mentioned above gives lower physical characteristics. It must be borne in mind that this range of silicon applies to a particular type of casting. The primary effect of the silicon is to decompose the combined carbon into graphite and ferrite ($Fe_3C = 3\ Fe + C$). Silicon is a vigorous graphitizer, and causes carbon to be precipitated during cooling through the critical range, being vigorous at high temperatures and decreasing as the temperature falls. Silicon promotes internal shrinkage and is a powerful deoxidizer. Its undesirable property of promoting internal shrinkage is detrimental to producing uniform results.

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[1] Metallurgist, National Brake and Electric Co., Milwaukee, Wis.

The substitution of a certain percentage of nickel to replace a part of the silicon greatly reduces internal shrinkage and is conducive of results which are very uniform. The use of nickel in reducing shrinkage is reflected in the pattern shrinkage. The untreated iron shows a shrinkage of $\frac{1}{4}$ in. per foot, but the same iron when nickel treated shows a shrinkage of only $\frac{3}{16}$ in. per foot.

TABLE I.—CHEMICAL COMPOSITION AND PHYSICAL PROPERTIES OF ALLOY CAST IRON.

	HEAT No. 1.	HEAT No. 2.	HEAT No. 3.	HEAT No. 4.	HEAT No. 5.	HEAT No. 6.
Silicon, per cent	1.65	1.64	1.58	1.41	1.68	1.66
Sulfur, per cent	0.073	0.077	0.090	0.096	0.073	0.074
Phosphorus, per cent	0.11	0.13	0.097	0.13	0.11	0.16
Manganese, per cent	0.91	0.95	0.77	0.88	1.13	1.17
Total Carbon	3.02	3.07	2.89	2.86	2.73	2.98
Nickel, per cent	0.75	1.03	1.21	0.97	0.98	1.02
Transverse Strength, lb. per sq. in	5100	5060	5390	5210	4900	5270
Deflection, in	0.21	0.19	0.187	0.24	0.17	0.24
Brinell Hardness	228	217	228	217	228	217
Tensile Strength, lb. per sq. in.	49 080	51 000	50 790	52 600	48 465	50 030

Nickel has the graphitizing property but to a less degree than silicon, its graphitizing influence being very mild when the iron reaches the upper critical range. It has an additional and valuable property of forming a solid solution with the ferrite, toughening the matrix with corresponding higher strengths.

In plain iron, the graphite is in the form of long flakes, their size depending upon the rate of cooling. Graphite in nickel-treated iron is very fine, uniformly distributed through the matrix and is of a rounded or nodular shape rather than in long sharp flakes. This produces additional strength, as the continuity of the matrix is not broken up to the same degree as in plain iron.

The percentage substitution of nickel for silicon in the particular type of castings with which the author has been working ranges from 1½ times the silicon deduction in heavy castings to 2½ times the deduction in the lighter castings. For example, a certain casting which formerly required 1.25 per cent of silicon was reduced to 0.75 per cent of silicon and 0.75 per cent of nickel substitute.

PHYSICAL PROPERTIES

The tensile strength of this material ranges from 55,000 to 60,000 lb. per sq. in., and can be uniformly obtained. The tensile strength is dependent upon the silicon, carbon and nickel content. Silicon and carbon substantially affect the results obtainable; as the percentage of either silicon or carbon is increased, the tensile strength is progressively reduced.

TABLE II.—COMPARISON OF PHYSICAL PROPERTIES AND CHEMICAL COMPOSITION OF TREATED AND UNTREATED IRON.

	UNTREATED	TREATED
Total Carbon, per cent	2.64	2.44
Manganese, per cent	0.24	0.739
Phosphorus, per cent	0.044	0.066
Silicon, per cent	1.120	1.24
Nickel, per cent	none	2.04
Tensile Strength, lb. per sq. in.	41 180	61 320
Brinell Hardness	302	163

The Brinell hardness on this material will usually be above 200. Although an iron that has been nickel treated will show a lower Brinell hardness than the same iron not nickel treated, the nickel-treated iron will machine freely, while iron that has not been nickel treated, of the same or even lower Brinell hardness, will be practically non-machinable.

The chemical composition and physical properties of several heats, taken at random from the files, are given in Table I.

The transverse strength on a standard A.S.T.M. arbitration bar will vary between 5400 and 6000 lb. per sq. in. Results as high as 6400 lb. per sq. in. have been obtained. The ratio of transverse strength to tensile strength on this material is consistently 1 to 10, that is, the tensile strength is very close to ten times the transverse strength.

The machineability of nickel-treated irons according to test on one type of castings of intricate design and various metal sections was

33 per cent better than on the same iron untreated. Another type of castings had sections so light that as cast with the usual mixture they always required grinding, as tool repair and breakage of castings made machining prohibitive. Two hundred of this type of casting were treated with nickel with the result that 199 were readily machined, only one breaking in the milling operation. An average of all castings on which machining operation data were taken by the author indicated 22 per cent better machining time. A check was made on the dressing of tools on one job and showed that only one-half the cutter grinding was necessary.

A comparison is shown in Table II of the physical properties together with chemical analysis of an iron treated with nickel and without nickel treatment.

PROCESS OF MANUFACTURE

The melting process is of prime importance, the most important factor being temperature, as low total carbon of the charge corresponds to a higher melting temperature consequently higher melting temperatures than those ordinarily used will be necessary for best results.

The most desirable stock charged for high steel mixtures to produce the most uniform results are coil springs, or coil springs and short rail ends. Experience and practice has proved that smaller charges than ordinarily used are conducive to better results:

CUPOLA DIAMETER, IN.	WEIGHT OF CHARGE, LB.
36	1000
48	2000
66	3000

To insure sufficient heat of combustion to produce the high temperature necessary, the fuel ratio, that is the ratio of coke to metal charges, should not be greater than 1 to 6½ or 7. Since the total carbon content depends largely on the melting rate, air volume and temperature, slightly higher blast pressures and consequently greater air volume must pass through the shaft of the cupola to stimulate faster melting. Recommended pressures and volumes are as follows:

CUPOLA DIAMETER, IN.	BLAST, OZ.	PRESSURE, INCHES OF WATER
24	3.92	7
36	7.85	14
48	8.96	16
60	10.08	18
72	10.64	19

The coke bed is also of vital importance and the following practice has proved to give good results. The initial coke bed of 40 to 50 in. above tuyeres is charged, ignited and burned to incandescence throughout, then about 6 in. more of coke is charged and metal and coke charged successively until 8 to 10 tons have been charged, depending upon the size of cupola. The stock is allowed to soak for at least one hour, preferably two. This soaking period heats the cupola and stock thoroughly and aids in increasing the melting rate of the first charge, also assuring good hot iron on the first tap.

In making ladle additions it is very desirable to preheat all alloy additions because of their high melting temperatures.

The following metal charge has been found to give very uniform results:

Metal charge in 36-in. cupola.—

1000 lb.	of coil springs
15 lb.	of 50-per-cent ferro-silicon

Ladle additions (per 100 lb. metal).—

12 oz.	of 70-per-cent ferro-manganese
8 oz.	of 50-per-cent ferro-silicon
24 oz.	of ferro-nickel shot.

The alloy addition is certainly beneficial if applied to a given class of work, taking into proper consideration coke bed, coke melt, air volume and the proper balance between total carbon, manganese, nickel, silicon and phosphorus. On the other hand, it may be stated that indiscriminate use of nickel or other alloys in the cast iron or semi-steel field is detrimental rather than beneficial.

Acknowledgments.—The author wishes to make acknowledgment of the assistance of F. B. Coyle, Metallurgist, New York Navy Yard, R. S. MacPherran, Chief Chemist, Allis Chalmers Manufacturing Co., and A. W. Lorenz, Metallurgist, Bucyrus Co.

DISCUSSION

Mr. T. R. Lawson.[1]—There are two questions I should like to ask. What is the relative cost of the treated as compared to the untreated material? Does the addition of the nickel affect the compressive strength? **Mr. Lawson.**

Mr. D. M. Houston.[2]—Mr. Coyle, formerly of the Brooklyn Navy Yard, has probably done more original work on combinations and introduction of silicon and manganese and nickel into liquid iron to obtain high strength than any one else, having supplied Mr. Greenhow with much of the data as well as the fundamental principles involved, and as he is here this afternoon I am wondering whether he could not answer this question? **Mr. Houston.**

Mr. F. B. Coyle.[3]—As regards the first question, the cost is very slightly higher than that of ordinary "semi-steel," say 25 per cent steel and the balance pig iron and returned scrap. As regards the second question, nickel increases the compressive strength. Out of a number of tests (the total carbon was about 2.90 to 3.20 per cent, nickel content, 1 per cent) the compressive strength of the material without nickel averaged 100,000 to 130,000 lb. per sq. in. With nickel it averaged 150,000 to 190,000 lb. per sq. in. **Mr. Coyle.**

Mr. R. S. MacPherran.[4]—The manufacture of this material is more or less past the experimental stage. One foundry is making a regular line of castings, the test bars showing a minimum tensile strength of 50,000 lb. per sq. in. Not quite as much nickel is used as Mr. Greenhow has used, but the bars run 50,000 to 55,000 lb. per sq. in. This foundry uses probably 90 per cent of steel with ferrosilicon and is making commercial castings regularly with this metal. **Mr. MacPherran.**

Mr. J. A. White.[5]—I should like to ask what is the proportion of combined carbon to this low total carbon in the cast iron. Of course that will vary somewhat with the section, but perhaps an average amount of combined carbon could be stated. **Mr. White.**

Mr. Coyle.—The combined carbon varies over quite a wide range, that is, the combined carbon in a cast iron possessing a full pearlitic structure. This can be more fully explained by reference **Mr. Coyle.**

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[1] Head, Department of Civil Engineering, Rensselaer Polytechnic Institute, Troy, N. Y.

[2] International Nickel Co., New York City.

[3] International Nickel Co., New York City.

[4] Chief Chemist, Allis-Chalmers Manufacturing Co., West Allis, Wis.

[5] Chemist and Metallurgist, Clark Equipment Co., Buchanan, Mich.

Mr. Coyle. to the accompanying Fig. 1. This constitutional diagram for cast iron was published by Maurer in *Kruppsche Monatschefte* in July, 1924. The ordinates represent percentage of total carbon and the abscissas percentage of silicon. The various areas represent the structure of cast iron of compositions included within that area when cast under the normal conditions for casting the A.S.T.M. standard arbitration test bar. However, by either accelerating or reducing the rate of cooling, the structure of an iron, the composition of which is included in any one area, may be made to correspond to that in any other area. In area I the structure is white or chilled, that is, the carbon is all combined and the constituents will be essentially iron carbide and pearlite. In area II*a*, the fracture will be mottled, or the components will be iron carbide, pearlite, and graphite. In area II the structure will consist of pearlite and graphite. In area II*b* the structure will be composed of pearlite, ferrite, and graphite (the pearlite predominating over the ferrite). In area III the structure will consist of the same phases as in area II*b* except that the ferrite will predominate over the pearlite. The broad extent of area II plainly indicates that there is no definitely fixed figure for the percentage of combined carbon in a pearlitic or eutectoid cast iron. In fact, I have records of analyses of pearlitic cast irons which show combined carbon varying from 0.50 to 1.20 per cent. The material described by Mr. Greenhow

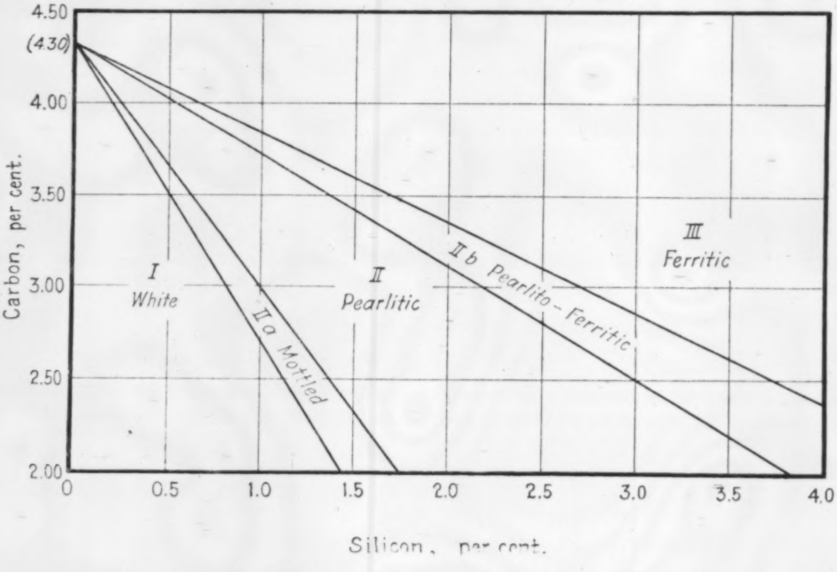

FIG. 1.—Constitutional Diagram for Cast Iron (Maurer).

Mr. Coyle.

consists of a full pearlitic structure with very fine well dispersed graphite.

In order to differentiate between gray iron and ordinary high test iron from the type of material described in Mr. Greenhow's paper reference to the accompanying Fig. 2 may be of some value. This is a modification of the Maurer diagram which is shown by the broken lines. Upon the Maurer diagram the speaker plotted the results of over four thousand tension tests of iron of a wide range of composition after assigning to each a designating mark signifying its inclusions within a certain range of tensile strength varying by 5000 lb. per sq. in. After these tests were all plotted it was possible to mark off the areas included by the solid lines. In the middle of the pearlite area of the Maurer diagram is an area N.P.T.S. included by four points: 3.00 per cent carbon and 1.00 per cent silicon, 3.15 per cent carbon and 1.30 per cent silicon, 2.70 per cent carbon and 1.80 per cent silicon, 2.50 per cent carbon and 1.35 per cent silicon. All compositions included within this area when cast under the conditions required for the A.S.T.M. standard arbitration test bar will give a tensile strength greater than 40,000 lb. per sq. in. However, there is a limit, and that limit is about 45,000 lb. per sq. in. One or two of the tests were as high as 46,200 lb. per sq. in., but the great majority were between 40,000 and 44,000 lb. per sq. in. This represents the

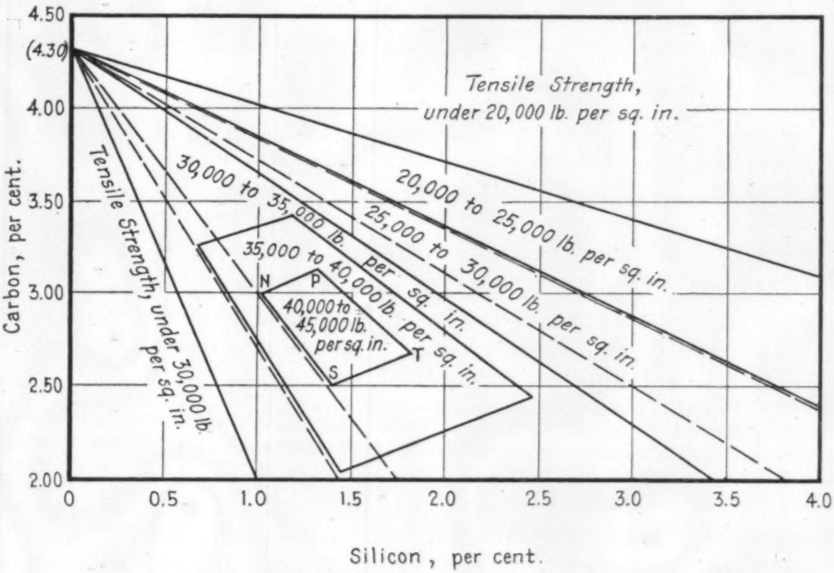

FIG. 2.—Constitutional Strength Diagram for Cast Iron (Coyle).

Mr. Coyle. maximum obtainable by ordinary means. To obtain greater strength it is necessary to resort to some special means as set forth in Mr. Greenhow's paper. Surrounding the area N.P.T.S. are other areas but all of lower strength. It was gratifying that this diagram after its construction had been checked exactly by twelve different individuals.

Mr. Greenhow. MR. M. E. GREENHOW[1] (*author's closure by letter*).—Replying to Mr. Lawson: The relative cost of nickel-treated iron compared with plain cast iron can be found by referring to a paper by Mr. D. M. Houston entitled "The Economic Use of Nickel in Cast Iron," much of the original cost data given therein having been furnished by the author of the present paper in connection with work carried on at the National Brake and Electric Co., Milwaukee, Wis. The cost of a nickel-treated iron based on the market cost of materials at the time showed that the use of nickel for several types of castings permitted the substitution of large percentages of scrap and steel for a part of the pig iron ordinarily used. For example, the usual 50 to 60 per cent was reduced to 18 to 25 per cent of the weight of the charge, giving at the same time better results than the higher pig iron percentages. One type of casting was made from all steel scrap with the proper addition of ferro-silicon, ferro-manganese and nickel.

The average increase in cost of nickel-treated iron for the several types of castings mentioned above was from $1.11 per net ton for 0.25-per-cent nickel content to $2.17 for 0.90-per-cent nickel content. This does not include any allowance for the alloy recovery from the gates and risers.

The effect of nickel on compressive strength has been satisfactorily answered by Mr. Coyle, who has made an exhaustive series of tests in this connection.

In reply to Mr. J. A. White: Although the combined carbon as stated by Mr. Coyle varies over a wide range, the author's experience has proved that for any particular type of casting with very close control of the chemical composition in the molten iron, temperature at time of pouring and consistent control of sands, ramming and percentage of moisture in the sand, the combined carbon results are surprisingly consistent.

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[1] Metallurgist, National Brake and Electric Co., Milwaukee, Wis.

TESTS OF THE ENDURANCE OF GRAY CAST IRON UNDER REPEATED STRESS

By H. F. Moore[1] and S. W. Lyon[2]

Synopsis

Tests of four gray cast irons are reported in this paper. The tests include: tension, compression, Charpy impact, Brinell hardness and rotating-beam fatigue tests. A new form of tension test specimen was used. The fatigue tests showed a fairly well marked endurance limit for each of the cast irons, but in each case the endurance limit was low, and the ratio of endurance limit to tensile strength was lower than is usually found for steel.

The fatigue strength of cast-iron test specimens was markedly increased by oft-repeated stress below the endurance limit.

Grooves in cast-iron test specimens reduced the endurance limit but a small amount. A much larger percentage of reduction of endurance limit by a similar groove has been found in the case of test specimens of steel and of copper.

Specimens of cast iron were tested under cycles of reversed flexure at temperatures ranging from room temperature to 1400° F. (760° C.). No great reduction of endurance limit was found for temperatures below 800° F. (425° C.). Above about 1200° F. (650° C.) the endurance limit was fully as high as was the tensile strength under prolonged steady load.

Specimens of cast iron tested under cycles of stress ranging from zero to a maximum gave an endurance limit 1.48 times the endurance limit for specimens ested under cycles of completely reversed flexural stress.

During the past year there have been made in the Materials Testing Laboratories of the University of Illinois a number of fatigue tests of specimens of gray cast iron. While the test results are too few to yield any comprehensive knowledge of the fatigue strength of cast iron, it is believed that the presentation of the data obtained will give some interesting information on a metal concerning whose fatigue strength very little is known.[3]

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[1] Research Professor of Engineering Materials, In Charge Investigation of the Fatigue of Metals, University of Illinois, Urbana, Ill.

[2] Engineer of Tests, Investigation of the Fatigue of Metals, University of Illinois, Urbana, Ill.

[3] A more extended account of these tests is given in University of Illinois Engineering Experiment Station, *Bulletin No. 164*, now in press.

CAST IRONS TESTED

Four different castings furnished the source of supply for test specimens, and the iron from each is designated by a laboratory number. The specimens of cast iron No. 91 were cut from a section of 6-in. centrifugally sand-cast pipe bought on the open market. The specimens of cast iron No. 92 were cut from a casting supplied by the Allis-Chalmers Co.; the casting was in the form of a cylinder 18 in. long and 12 in. in inside diameter, with walls 1 in. thick. The specimens of cast iron No. 93 were cut from a similar cylindrical casting with walls 3¼ in. thick. The specimens of cast iron No. 94 were cut from the inner wall of a double-walled cylinder casting weighing about 25 tons.

The chemical analyses of the irons tested are given in Table I. All the irons were tested as cast; no special heat treatment was used either at the foundry or at the laboratory. Cast iron No. 91 was allowed to cool in the mold, not shaken out hot as is frequently the case with centrifugal-cast pipe.

TABLE I.—CHEMICAL COMPOSITION OF CAST IRONS TESTED.

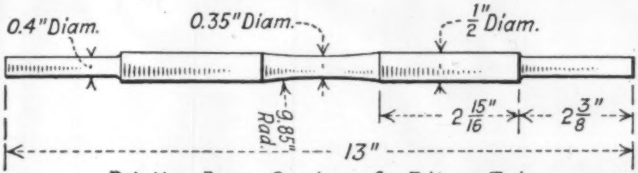
Cast Iron	Silicon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Graphitic Carbon, per cent	Combined Carbon, per cent	Total Carbon, per cent
No. 91	1.42	0.32	0.75	0.065	2.72	0.84	3.56
No. 92	1.10	0.62	0.51	0.093	2.76	0.68	3.44
No. 93 A	1.10	0.63	0.51	0.095	2.80	0.55	3.35
No. 93 B	1.10	0.59	0.51	0.094	2.78	0.57	3.35
No. 93 C	1.10	0.60	0.46	0.096	2.82	0.43	3.25
No. 94 A	1.16	0.58	0.38	0.102	....[a]	....[a]	3.32
No. 94 B	1.14	0.61	0.38	0.100	....[a]	....[a]	3.30
No. 94 C	1.13	0.57	0.40	0.103	....[a]	....[a]	3.30

[a] Combined carbon was less than 0.05 per cent; nearly all carbon was graphitic.

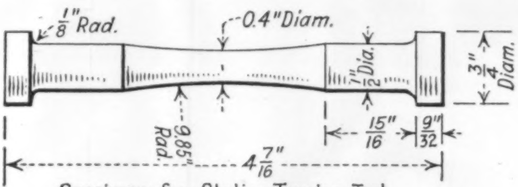
Micrographs of the irons tested show no unusual features. It will be noted from Table I that cast iron No. 94 has nearly all its carbon in the form of graphite. Micrographs of etched specimens of this iron showed, as might be expected, less pearlite than did micrographs of etched specimens of irons Nos. 91, 92 and 93.

TEST SPECIMENS AND METHODS OF TESTING

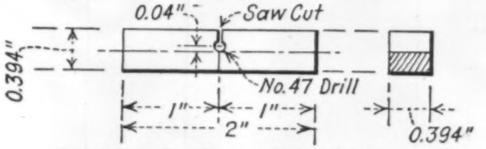
From each cylindrical casting furnished there were cut strips with axes parallel to the axis of the cylinder. For cast irons Nos. 93 and 94, strips were cut from three concentric layers. Strips cut from the outer layer were marked *A;* strips from the middle layer, *B;* and strips from the inner layer, *C*. From these strips, test specimens were machined of the form and size shown in Fig. 1.[1] Especial attention is called to the tension-test specimens, which were designed so insure a uniform distribution of stress across the critical cross-section. To insure axial loading they were held in Robertson ball-teated shackles.[2]



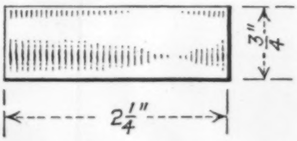
Rotating-Beam Specimen for Fatigue Test.



Specimen for Static Tension Test.



Specimen for Charpy Impact Test.



Specimen for Static Compression Test.

FIG. 1.—Test Specimens.

Tension tests, compression tests, Charpy impact tests and Brinell hardness tests were made using standard machines. In the Brinell tests a load of 1000 kg. was used, the ends of broken Charpy specimens serving as Brinell test specimens. Fatigue tests, except tests under alternating flexure combined with steady tension, were made on rotating-beam testing machines of the Sondericker (or Farmer) type, which has been described several times before the Society.[3]

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[1] For specimens of cast iron No. 91 the thin walls of the pipe made necessary some reduction in dimensions for some specimens.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part I, p. 857 (1925); also 1926 Book of A.S.T.M. Tentative Standards, p. 957, Fig. 6.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. VII, p. 252 (1907); Vol. XIX, Part II, p. 710 (1919); Vol. 22, Part II, p. 267 (1922); Vol. 25, Part II, p. 70 (1925).

GENERAL TEST RESULTS

Table II gives the general results of the static tension tests, the static compression tests, the Charpy tests, the Brinell tests and the rotating-beam fatigue tests. In the tension tests there was no clearly marked elastic limit and no detectable yield point, and no values for elastic limit or for yield point have been reported.

TABLE II.—RESULTS OF TENSION TESTS, COMPRESSION TESTS, CHARPY IMPACT TESTS, BRINELL HARDNESS TESTS AND FATIGUE TESTS OF CAST IRON.

All fatigue tests were made on rotating-beam testing machines.

Cast Iron	Tensile Strength, lb. per sq. in.	Compressive Strength, lb. per sq. in.	Charpy Tests (notched-bar), ft-lb.	Brinell Hardness Number	Fatigue Tests	
					Endurance Limit, lb. per sq. in.	Endurance Ratio[1]
No. 91...........	26 200	96 000	4.0	162	12 000	0.46
No. 92...........	31 600	111 000	11.8	148	10 500	0.33
No. 93 A.........	28 100	94 000	2.7	138	10 000	0.36
No. 93 B.........	25 300	85 000	2.2	132	9 000	0.36
No. 93 C.........	28 000	87 300	2.5	139	10 000	0.36
No. 94 A.........	21 400	82 500	1.6	89	7 000	0.33
No. 94 B.........	20 700	83 500	1.6	91	7 200	0.35
No. 94 C.........	20 500	82 000	1.5	88	7 800	0.38

[1] The endurance ratio is the ratio of endurance limit to tensile strength.

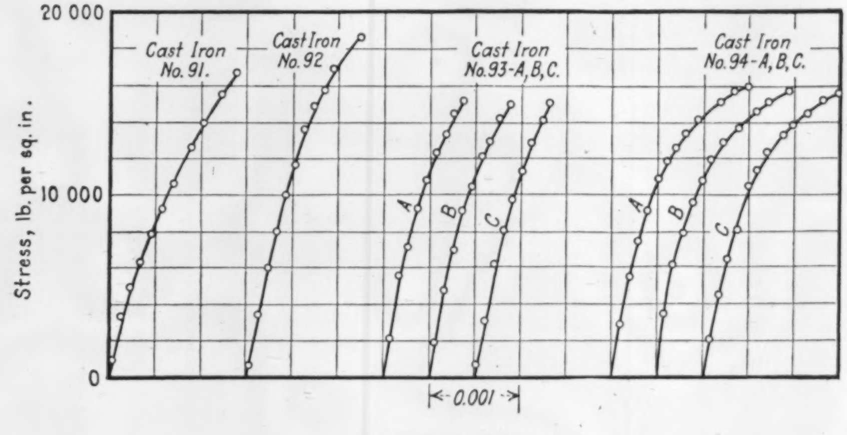

FIG. 2.—Stress-Strain Diagrams for Tension Tests at Room Temperature.

Figure 2 gives typical stress-strain diagrams for tension tests at room temperature and Fig. 3 gives typical $S-N$ diagrams for fatigue tests. As indicated by the $S-N$ diagrams, the endurance limit

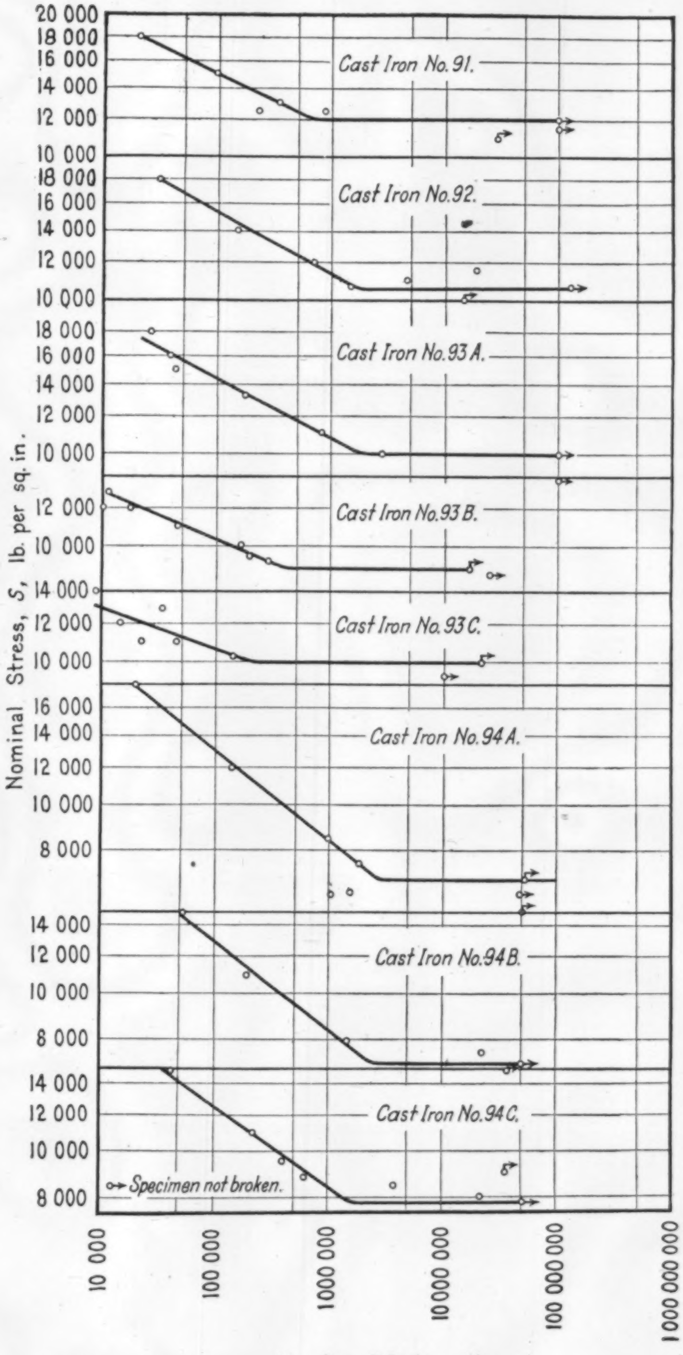

FIG. 3.—$S-N$ (Stress-Cycle) Diagrams for Rotating-Beam Fatigue Tests of Cast-Iron.

for each of the irons tested is fairly well marked but is low, and the endurance ratio[1] is also low.

RESULTS OF SPECIAL TESTS

Effect of "Understressing" Cast Iron.—Several series of special tests were run on one or more of the irons. One series, run on irons Nos. 93 and 94, was a study of the effect of "understressing" on the fatigue strength of cast iron. "Understressing" consists of subjecting specimens of cast iron to several millions of cycles of stress just below the endurance limit of the virgin metal.

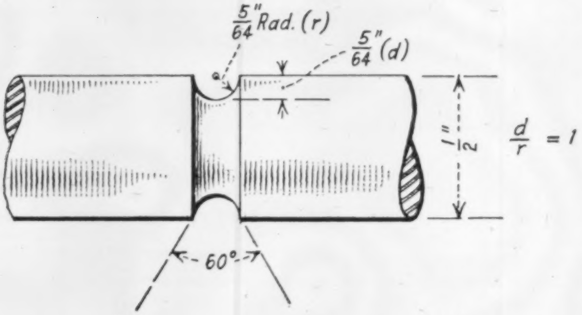

FIG. 4.—Groove in Specimens for Studying Effect of a "Stress Raiser."

Several experimenters have found[2,3,4,5] that such understressing appreciably raises the fatigue strength of rolled steel, but it has been thought that it would have little or no effect on the fatigue strength of a brittle material like cast iron. However, the tests showed a very distinct increase in endurance limit of the understressed specimens of cast iron, the increase amounting to 43 per cent of the endurance limit of the virgin metal in the case of cast iron No. 94.

This unexpected result suggests that only a very small amount of elongation may be necessary to cause effective adjustment of a metal to repeated strain below the endurance limit, and that elongation and reduction of area, as shown by a tension test, are not reliable indices of such adjustability. An illustration of this latter tentative conclusion is furnished by fatigue tests of specimens of brass, a metal which shows far higher ductility (as measured by elongation and reduction of area) than does cast iron, but which shows only a 6 per cent increase of endurance limit upon understressing. The question is raised in the minds of the authors whether there may not be a valuable property of metals, which may be called "strain adjustability," and which is quite distinct from ductility.

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[1] The endurance ratio is the ratio of endurance limit to tensile strength.

[2] R. R. Moore, ***Proceedings***, Am. Soc. Testing Mats., Vol. 24, Part II, p. 565 (1924); ***Bulletin No. 142***, p. 27, Engineering Experiment Station, University of Illinois, Urbana, Ill.

[3] H. W. Gillett and E. L. Mack, ***Proceedings***, Am. Soc. Testing Mats., Vol. 24, Part II, p. 490 (1924).

[4] H. J. Gough, "The Fatigue of Metals" (Scott Greenwood & Son—Van Nostrand), p. 108.

[5] J. H. Smith, ***Journal*** (British) Iron and Steel Inst., 1910, Part II, p. 280.

Effect of Grooves on the Fatigue Strength of Cast Iron.—Fatigue tests on specimens of rolled steel have shown that abrupt changes in form, such as holes and grooves, markedly reduce the fatigue strength of a specimen or of a machine part, but that the reduction of strength is not so great as is indicated by the determination of the theoretical localized stress at the hole or the groove. A common explanation of this discrepancy has been that the ductility of the metal allows redistribution of stress around the hole or the groove before a fatigue crack is formed. It has been thought that brittle metals would be more weakened by grooves and holes than would ductile metals.

TABLE III.—EFFECTIVE REDUCTION OF FATIGUE STRENGTH BY GROOVE.

See Fig. 1 for dimensions of standard specimen and Fig. 4 for dimensions of specimen with groove.

Material	Endurance Limit, lb. per sq. in.		Reduction of Endurance Limit due to Groove, per cent	Elongation in 2 in., per cent
	Standard Specimen	Specimen with Groove		
Griffith formula	.....	.....	67	....
1.02 per cent carbon steel (spring steel), water quenched	95 000	55 000	42	....
Annealed copper	10 000	6 000	40	56.4
3.5 per cent nickel steel, oil quenched, drawn at 1100° F. (595° C.)	64 000	40 000	37	22.6
Chrome-nickel steel, oil quenched, no draw	85 000	59 000	35	....
1.02 per cent carbon steel (spring steel), oil quenched, no draw	85 000	59 000	31	....
0.49 per cent carbon steel, normalized	33 000	24 000	27	26.5
95–5 bronze, annealed	23 000	20 500	7	66.9
Cast iron No. 92	10 000	9 700	3	less than 1.0

A series of experiments on the effect of a groove on the fatigue strength of cast iron and of other metals has been partly completed. The groove was of the form and size shown in Fig. 4. The effect of a groove for cast iron was studied experimentally by Mr. N. P. Inglis, using specimens made of cast iron No. 92, while a study of the effect of a groove on other metals is now being made by Mr. H. E. Degler. The theoretical stress-intensification caused by the groove shown in Fig. 4 would result in a stress 3.0 times the stress computed by the ordinary flexure formula.[1] The reduction of strength due to this stress-intensification might then be expected to be 66.7 per cent. The effective reductions for various metals were found to be as given in Table III. The value of the reduction for any metal was determined from a comparison of the endurance limit for standard specimens (Fig. 1) and the nominal endurance limit for specimens with groove (Fig. 4).

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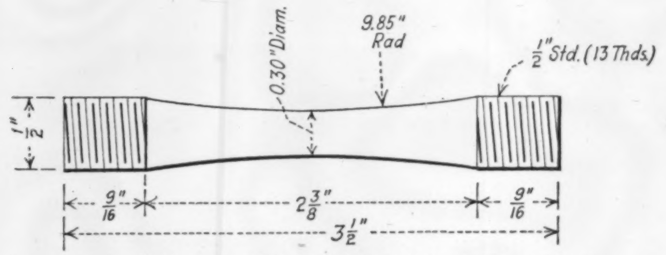
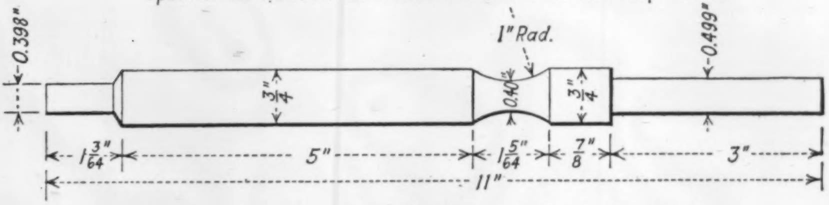
[1] Computed from the formula derived by A. A. Griffith from experiments with his soap film apparatus, see "Effect of Scratches on the Strength of Shafts," Advisory Committee for Aeronautics (British), December, 1918.

Evidently, the magnitude of effect of groove is no direct function of brittleness. The brittlest of the metals, cast iron, shows the least reduction of strength due to groove, while the second most ductile, annealed copper, shows the second greatest reduction. It is to be noted that the metals which are either very fine grained or are of a high degree of homogeneity, seem to be worse affected by a groove than do the coarser grained and less homogeneous metals. The following tentative explanation is suggested by the writers: The "fine" steels and the annealed copper approach more nearly than do cast iron, bronze, and medium steel to the conditions of homogeneity and of infinite divisibility, which are assumed as the basis for all our stress formulas. The groove reduces the fatigue strength of the metal, but the *theoretical* reduction is from the strength of an ideal material, one which fulfills the assumptions of the theory of elasticity. The *effective* reduction is from a lesser strength, the strength of an actual material. The degree in which any metal approaches the ideal condition of homogeneity and divisibility may, perhaps, be measured



Specimen for Static Tension Tests of Cast Iron at Elevated Temperatures.



Specimen for Fatigue Tests of Cast Iron at Elevated Temperatures.

FIG. 5.—Specimen of Cast Iron for Fatigue Tests at Elevated Temperatures.

by the proportional damage done by a groove or other "stress raiser." A cruder way of putting this idea is as follows: A piece of steel with many minute holes in it has its strength but little affected if another small hole is bored in it; a piece of steel without holes is much more affected by the boring of a single hole. Perhaps we can go so far as to quote the homely proverb, "You can't spoil a bad egg."

Tests of Cast Iron at Elevated Temperatures.—A series of static tension tests and fatigue tests was made on cast iron No. 92 at various temperatures. The maximum temperature was 1400° F. (760° C.). The tension tests were made on a 10,000-lb. four-screw Olsen testing machine with an electric furnace attached. The temperature was measured by a thermocouple making contact with the specimen at its critical section. The fatigue tests were made on machines of the rotating spring type with electric furnaces attached.[1] The specimens for static tension tests and for fatigue tests at elevated temperatures are shown in Fig. 5.

TABLE IV.—RESULTS OF STATIC TESTS AND FATIGUE TESTS OF CAST IRON AT ELEVATED TEMPERATURES.

Temperature		Brinell Hardness Number	Tensile Strength (ordinary rate of loading), lb. per sq. in.	Tensile Strength (prolonged test), lb. per sq. in.	Endurance Limit, lb. per sq. in.
deg. Fahr.	deg. Cent.				
70	20	148	30 000	30 500	12 000
200	95	154	.....	.....	.....
400	205	145	30 500	28 400	.....
600	315	149	.....	.....	.....
800	425	154	29 600	24 900	.....
1000	540	135	25 600	11 800	10 200
1100	595	115	.....	.....	.....
1200	650	60	20 300	7 800	.....
1400	760	32	11 300	3 500	6 500

Brinell tests were made at various temperatures. The Brinell specimen, $\frac{3}{4}$ in. square by $\frac{3}{8}$ in. thick, was placed inside an electric furnace on a block supported on the weighing table of the 10,000-lb. testing machine, and the temperature brought up to and held at the desired value for a period of at least 15 minutes. The 10-mm. Brinell ball, which was mounted on the end of a long bar attached to the moving head of the testing machine, was run down into the furnace against the specimen and the impression obtained at the desired load. The load applied to the ball was 1500 kg. except for the tests at 1200° F. (650° C.) and 1400° F. (760° C.), where 500 kg. was used, thereby keeping the diameter of impression within a range between

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[1] The arrangement of apparatus for static tension tests is similar to that shown in the article by V. T. Malcolm, *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 36 (1924), and the testing machine for fatigue tests is similar to that shown on page 46 of the same article, with the addition of an electric furnace around the reduced section of the specimen. Fuller details of testing machines are given in *Bulletin No. 152*, Engineering Experiment Station, University of Illinois, Urbana, Ill.

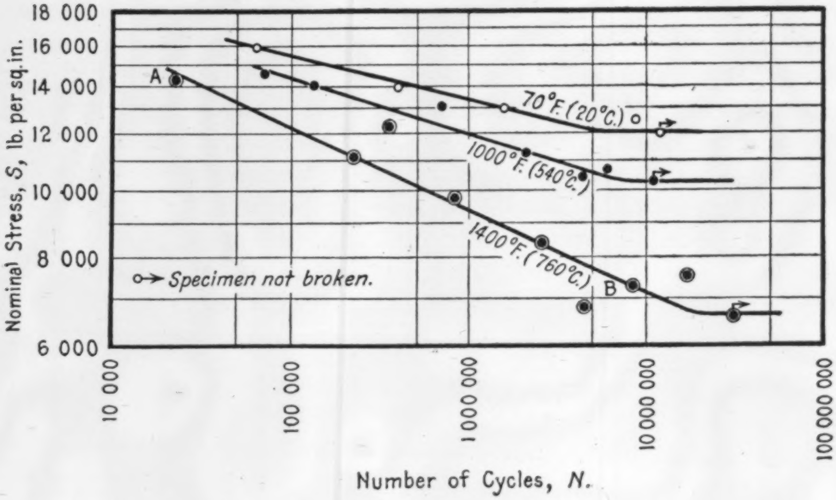

FIG. 6.—$S-N$ (Stress-Cycle) Diagram for Fatigue Tests at Elevated Temperatures.

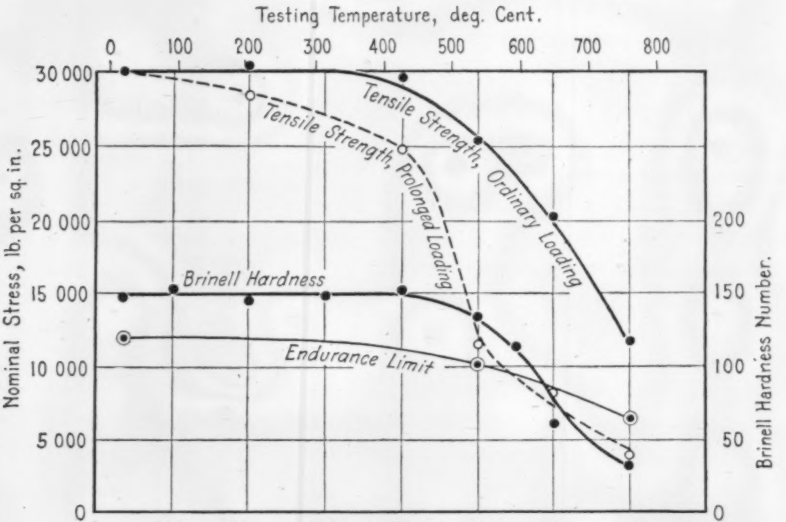

FIG. 7.—Results of Tests at Elevated Temperatures.

3.1 and 4.1 mm. In all tests the pressure was maintained for 30 seconds.

Two series of static tension tests were made. One series was carried out at ordinary speeds of testing while the other was performed at a much slower rate, each test lasting several days. These tests are called "prolonged" tests, and while they were not sufficiently prolonged to give the strength of cast iron under indefinitely long steady loading, they do furnish an interesting comparison with test results at ordinary speeds. In these prolonged tests, no increment of load was added to a specimen until the strain under the previous load had ceased to show appreciable increase. Indication of appreciable increase of strain was given by the drop of the beam of the testing machine and, in some of the tests, by increase of extensometer readings. When the beam of the testing machine dropped it was immediately brought up to balance and the load maintained. This was done repeatedly until a time was reached when the beam remained stationary or fell only after an interval of several hours.

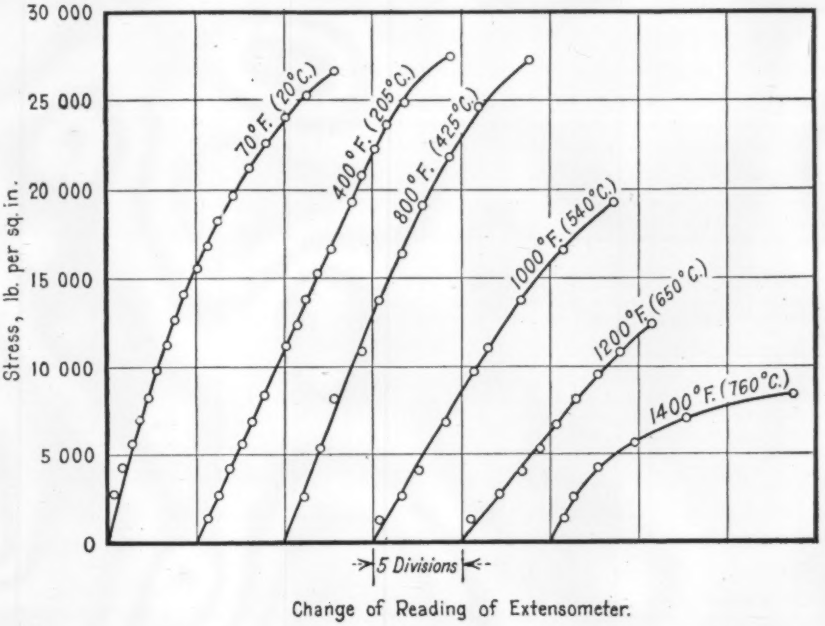

FIG. 8.—Stress-Strain Diagrams for (Static) Tension Tests at Elevated Temperatures.

Table IV gives the summarized results of all the tests made at elevated temperatures. Figure 6 gives the $S-N$ diagrams for the fatigue tests at elevated temperatures, and Fig. 7 is a graphical summary of the test results. Figure 8 shows typical stress-strain[1] diagrams for the ordinary rate of loading tension tests at elevated temperatures.

The results of the ordinary static tests at high temperatures are in good agreement with previous work on cast iron by Harper and MacPherran,[2] Bach,[3] and J. E. Howard.[4] The results show that the cast iron tested maintains its tensile strength practically constant up to a temperature of about 800° F. (425° C.), but that the drop in strength at higher temperature is rapid. The stress-strain diagrams (Fig. 8) show the general character of the changes in the stress-strain relationship with increasing temperature. It is to be noted that there is little change in the general slopes of the curves up to 800° F. (425° C.), but that above this temperature a decided change is in evidence. At 400 and 800° F. (205 and 425° C.), the relationship approaches the straight line law.

TABLE V.—ENDURANCE LIMITS FOR SPECIMENS OF CAST IRON NO. 92 UNDER DIFFERENT RANGES OF STRESS.

	Symbol	Value
Endurance limit under reversed flexure (range ratio = −1.0), lb. per sq. in.	S_{-1}	10 500
Endurance limit under stress range from zero to maximum tension (range ratio = 0), lb. per sq. in.	S'_0	15 500
Endurance limit under stress range from zero to maximum compression (range ratio = 0), lb. per sq. in.	S''_0	65 000
Tensile strength, lb. per sq. in.	S'	31 600
Compressive strength, lb. per sq. in.	S''_u	111 000
Endurance ratio for tension	S'_0/S'_u	0.44
Endurance ratio for compression	S''_0/S''_u	0.59
Endurance ratio for alternating stress	S_{-1}/S'_u	0.33
....	S'_0/S_{-1}	1.48

Referring again to Fig. 7, it may be seen that the curve of hardness-temperature follows the tensile strength - temperature curve fairly well up to 1000° F. (540° C.), after which the drop of the former is more gradual than the latter.

The results of the prolonged static tension tests (Fig. 7) show that at normal temperature the tensile strength of cast iron is not affected by the variations in rate of loading, which ranged from a very small value for the prolonged tests to about 2,000 lb. per sq. in. per second for the ordinary tests. At higher temperatures the difference in tensile strength due to rate of loading is most marked.

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[1] The extensometer was attached to the specimen holders and the extensometer readings do not measure precise strain in the specimen, although they do furnish data for showing the general shape of the stress-strain diagrams.

[2] Harper and MacPherran, "Tensile Tests of Cast Iron at Various Temperatures," *Iron Age*, Vol. 110, p. 793.

[3] C. Bach, "Elastizität und Festigkeit," 1920, p. 180.

[4] J. E. Howard, "Physical Properties of Iron and Steel at High Temperature," *Iron Age*, Vol. 45.

It is to be noted that the prolonged tests, while they give some interesting comparisons with tension tests at ordinary speed, do not determine the tensile strength of cast iron subjected to high temperatures for periods of months or years.

The fatigue tests at elevated temperatures bring out several interesting points, especially the fact that at very high temperatures the fatigue limit for the cast iron tested seems to be greater than the tensile strength under prolonged loading. Similar results were obtained on other materials at the University of Illinois[1] and by Tapsell and Bradley.[2] It should, of course, be pointed out that there is a greater departure from Hooke's law at higher temperatures (Fig. 8) and consequently a greater difference between the actual and the computed stress in the fatigue specimen.

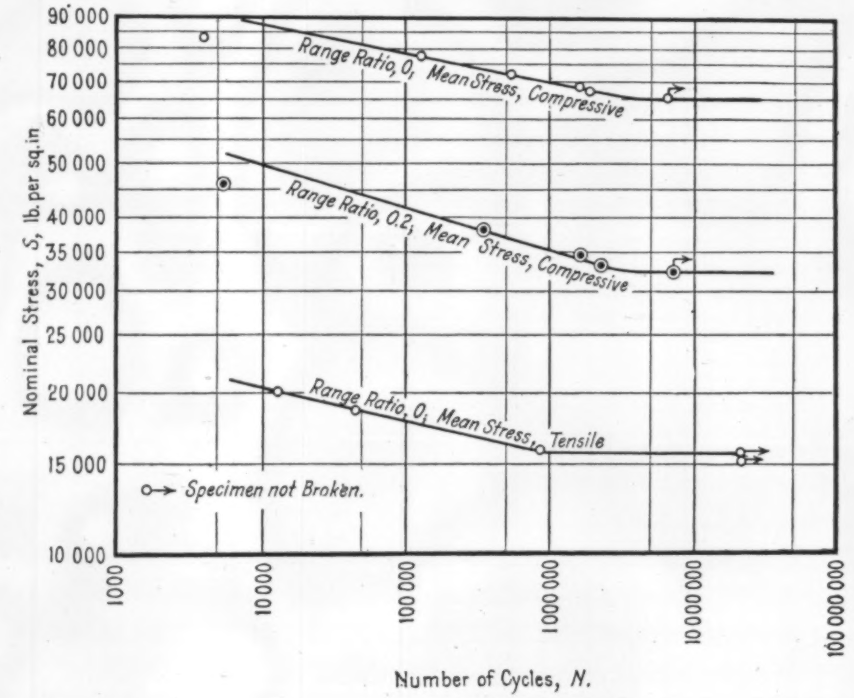

FIG. 9.—$S-N$ (Stress-Cycle) Diagrams for Fatigue Tests with Varying Range of Stress.

An examination of the $S-N$ diagrams in Fig. 6 shows that the "knees" of the curves at high temperatures occur at a greater number of cycles than is usually the case. An explanation of this may be offered, based on the work of Dickenson[1] and others which indicates that at higher temperatures metals behave as viscous fluids, and the cause of failure after a considerable period at elevated temperature may be in part due to the internal stress gradually set up by viscous flow.

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[1] *Bulletin No. 152*, Engineering Experiment Station, University of Illinois, Urbana, Ill., p. 22.

[2] *Journal* (British) Institute of Metals, Vol. XXXV, No. 1, p. 75 (1926).

Upon consulting Fig. 7 it will be seen that the curve of endurance limits follows the curve of tensile strengths fairly closely with but little change until 1000° F. (540° C.) is reached, after which the latter curve falls off more rapidly. This condition is best illustrated by a comparison of the endurance ratios at three temperatures. At 70° F. (20° C.) the value is 0.400, at 1000° F. (540° C.) it is also 0.400, and at 1400° F. (760° C.) it is 0.574.

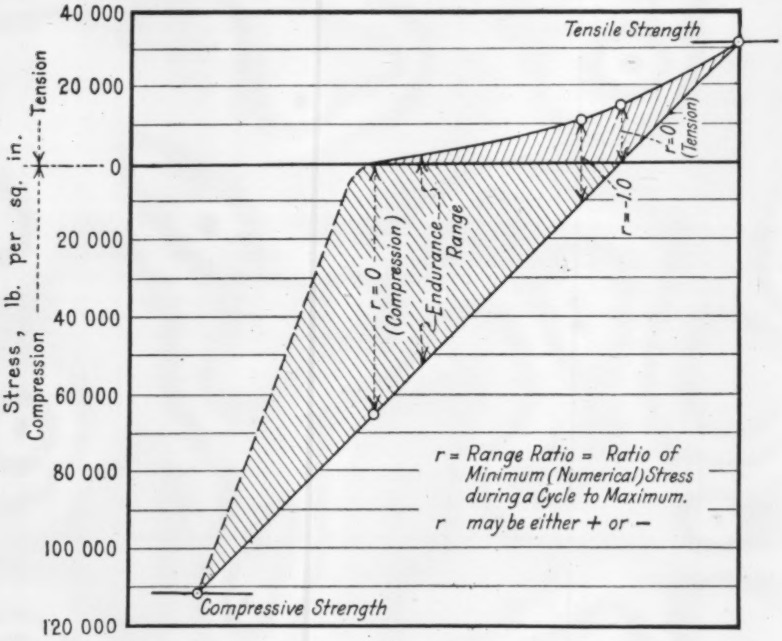

FIG. 10.—Modified Goodman Diagram for Fatigue Test Results with Varying Range of Stress.

Fatigue Tests of Cast Iron under Cycles of Stress Ranging from Zero to a Maximum.—A few tests of specimens of cast iron No. 92 were made to determine the endurance limit for cycles of stress ranging from zero to a maximum. One series of tests was made by superimposing a steady tensile stress on a specimen subjected to cycles of reversed flexure,[1] and a second series of tests was made on specimens subjected to cycles of axial stress varying from zero to a maximum in compression.[1]

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[1] J. H. S. Dickenson, "The Flow of Steels at a Red Heat," *Engineering* (London), Vol. 114, p. 326 (1922).

Figure 9 shows the $S - N$ diagrams. The values for endurance limit are given in Table V. It is to be noted that the ratio of endurance limit for cycles of stress varying from zero to a maximum in flexure (range ratio = 0)[2] is 1.48 times the endurance limit for cycles of completely reversed flexure (range ratio $= -1$). This result is not widely different from that found in tests of steel.

The general results for the tests are plotted in Fig. 10, following the general plan of the well-known Goodman diagram for range of stress, but extending the diagram into the field of cycles of compressive stress. The part of the diagram in broken lines is extrapolated beyond the test data.

It is to be noted that these results on effect of range of stress on fatigue strength of cast iron are very few and are only tentative. They are published only because of the fact that very few, if any, data are available for the study of this subject.

Acknowledgment.—Acknowledgment is made to the Allis-Chalmers Manufacturing Co., whose financial support made possible these tests, and particularly to Messrs. J. Fletcher Harper, R. S. MacPherran and E. H. Brown of that company. Acknowledgment is made to Messrs. N. J. Alleman and C. T. Han of the laboratory force of the Investigation of the Fatigue of Metals. Especial acknowledgment is made to Mr. N. P. Inglis of Liverpool, England, Visiting Research Fellow at the University of Illinois, to whom much of the credit for planning and carrying out the special tests mentioned is due.

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[1] Testing machines used are described in *Bulletins Nos. 142* and *152*, Engineering Experiment Station, University of Illinois, Urbana, Ill.

[2] The term "range ratio" is used to denote the ratio of the minimum (numerical) stress during a cycle to the maximum.

[For Discussion on Fatigue of Metals, see page 134.—ED.]

CORROSION-FATIGUE OF NON-FERROUS METALS[1]

By D. J. McAdam, Jr.[2]

Synopsis

Part I gives a brief resumé of previous work, an outline of the investigation and a description of the material and the methods of investigation.

Part II discusses corrosion-fatigue of nickel-copper alloys. The following subjects are considered: Fatigue and corrosion-fatigue graphs for nickel, monel metal, other nickel-copper alloys and copper; complexity of some corrosion-fatigue graphs; effects of cold-working on corrosion-fatigue limits; relationship between chemical composition and corrosion-fatigue limits of nickel-copper alloys.

Part III discusses corrosion-fatigue of aluminum alloys. The alloys considered are aluminum, aluminum-manganese alloy and duralumin. Fatigue and corrosion-fatigue graphs for these alloys are described and discussed. Effects of cold-working and heat-treatment of aluminum alloys are discussed.

Part IV is a general discussion of corrosion-fatigue of metals. Tentative hypotheses are discussed under the following headings: Intrinsic corrosion-fatigue limits; secondary influences affecting the corrosion-fatigue limit, relationship between fatigue and corrosion-fatigue.

PART I.—OUTLINE OF INVESTIGATION, DESCRIPTION OF MATERIAL AND METHODS

Previous Investigation:

Results of a previous investigation of corrosion-fatigue of steel were presented at the Annual Meeting, June, 1926.**(1)**[3] A later paper,**(2)** presented at the annual meeting of the American Society for Steel Treating, September, 1926, gave results of continued investigation of corrosion-fatigue of steel.

As described in these two papers, rotating-cantilever specimens, of the conically tapered design described in former papers,**(5,6,7)** were subjected to simultaneous fatigue and corrosion. For this purpose a stream of either fresh or salt water was diagonally applied so as to sweep the specimen from the outer to the inner fillet and surround the stressed surface entirely by water. The investigation included carbon steels, ordinary alloy steels, and "corrosion-resistant" steels.

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[1] Published by permission of the Secretary of the Navy.

[2] Metallurgist, U. S. Naval Engineering Experiment Station, Annapolis, Md.

[3] The boldface numbers in parentheses refer to the papers given in the list of references appended hereto.

The experiments showed that even slight corrosion simultaneous with fatigue may cause failure at stresses far below the ordinary "endurance limit." It was also shown that severe corrosion prior to fatigue lowers fatigue resistance much less than slight corrosion simultaneous with fatigue. The simultaneous influence of corrosion and fatigue may therefore, be called "corrosion-fatigue." Under corrosion-fatigue the stress-cycle graph for steel approaches a horizontal asymptote which may be called a "corrosion-fatigue limit."

It was also shown that for any one corrosive agent the corrosion-fatigue limit depends on the corrosion resistance of the steel, but apparently depends little if at all on the physical properties as determined by an ordinary tension test, with the possible exception of the modulus of elasticity.

In closing the discussion of the first paper, reference was given to a paper on "Fatigue of Brasses" by B. P. Haigh(3) published in 1917. In some experiments described in Haigh's paper, brasses and bronzes were subjected to alternating stress while in contact with corrosive agents such as ammonia, salt water and hydrochloric acid. He found slight lowering of the stress-cycle graph when Muntz metal and Naval Brass were etched with ammonia, but found no such lowering with the other combinations of metal and corrosive agent. Haigh's experiments were extended to only about 1,500,000 cycles, so a corrosion-fatigue limit for brass and ammonia was not obtained. Haigh ascribed the effect on brass to the well-known strong attack of ammonia on the Beta constituent of brass. He points out that the damaging effect of ammonia is not produced unless corrosion and fatigue are simultaneous.

Although Haigh suggested the possibility that corrosion may affect unfavorably the endurance properties of metals in service, his results gave no indication of the great and general effects of even slight corrosion when simultaneous with fatigue. Haigh's experiments, however, made less impression than they deserved. Seven years later Gough's comprehensive book, "The Fatigue of Metals," made no mention of corrosion. Haigh's experiments had escaped my attention, until after the first two papers(1,2) were published. The absence of any reference to corrosion in Gough's 1924 book(4) led me to believe that no important investigation of corrosion-fatigue had been made.

Outline of Continued Investigation:

Preliminary investigation of corrosion-fatigue of non-ferrous metals at the Naval Engineering Experiment Station has now progressed far enough to permit presentation of some results obtained with nickel, copper, nickel-copper alloys, aluminum and aluminum alloys. For this investigation the International Nickel Co. supplied nickel and monel metal, the American Nickel Co. supplied a nickel-copper alloy having about 50 per cent nickel, the Aluminum Co. of America supplied aluminum alloys, and the copper and cupro-nickel were obtained from the American Brass Co. The nickel and monel metal were supplied in the form of 1-in. square bars. The other nickel-copper alloys and the copper were supplied in the form of 1-in. round bars. The aluminum alloys were supplied in the form of $1\frac{1}{8}$-in. round bars.

Table I.—Chemical Composition of Material.

All Values are Averages of at Least Four Determinations

Material	Designation	Nickel, per cent	Copper, per cent	Aluminum, per cent	Iron, per cent	Manganese, per cent	Magnesium, per cent	Silicon, per cent	Carbon, per cent	Phosphorus, per cent	Sulfur, per cent
Nickel, Cold-Rolled......	EO	98.96[a]	0.17	.....	0.57	0.03	.....	0.09	0.098	0.006	0.011
Monel Metal, Cold-Rolled	EP	67.51[a]	29.54	.....	1.76	0.95	.....	0.05	0.159	0.015	0.017
Nickel-Copper Alloy, Cold-Rolled..........	EI	48.33[a]	48.37	.....	1.12	1.90	.....	0.05	0.23	.....	.....
Nickel-Copper Alloy, Cold-Worked.........	HE	21.24[a]	77.92	.....	0.51	0.29	.....	0.005	0.031	0.007	0.011
Copper, Electrolytic, Cold-Worked.........	HF	.....	99.996[a]	.....	0.004	.....	.....	.....	.....	.....	.....
Copper, Electrolytic, Hot-Rolled...........	EE	.....	99.996[a]	.....	0.004	.....	.....	.....	.....	.....	.....
Aluminum.............	DR	.....	0.12	99.35[a]	0.30	0.005	None detected	0.22	.....	.....	.....
Aluminum-Manganese Alloy...............	DS	.....	0.16	97.98[a]	0.29	1.22	None detected	0.35	.....	.....	.....
Duralumin.............	GT	.....	3.72	94.06[a]	0.42	0.50	0.28	1.02	.....	.....	.....
Duralumin.............	DU	.....	4.18	93.89[a]	0.54	0.60	0.42	0.37	.....	.....	.....

[a] Obtained by difference.

These materials in various conditions as regards cold-working and heat-treatment have been subjected to corrosion-fatigue in fresh and salt water.

Machines and Specimens:

The rotating-cantilever machines and specimens have been described in previous papers.(5,6,7) The conically tapered specimen is so designed that the maximum stress is $\frac{3}{4}$ in. out from the inner fillet, and the stress varies only about $1\frac{1}{2}$ per cent over a length of $1\frac{1}{2}$ in. With this specimen, therefore, a comparatively large region may be subjected to corrosion-fatigue.

The method of alternate longitudinal and transverse polishing of this specimen has been previously described.(6) The surface finish by this method is sufficiently smooth to permit examination of the microstructure at 100 magnification. Details of machining and polishing are tabulated and kept on file at the Naval Engineering Experiment Station. This information is available for those who are interested.

Table II.—Heat Treatment of Material.

Material	Designation	By Naval Engineering Experiment Station				By Manufacturer
		Heated to		Time Held, minutes	Cooled in	
		deg. Fahr.	deg. Cent.			
Nickel, Cold-Rolled	EO-6	600	316	120	Furnace	
Nickel, Cold-Rolled	EO-14	1400	760	60	Furnace	
Monel Metal, Cold-Rolled	EP-8	800	427	180	Furnace	
Monel Metal, Cold-Rolled	EP-14	1400	760	60	Furnace	
Nickel-Copper Alloy, Cold-Rolled	EI	As received				
Nickel-Copper Alloy, Cold-Rolled	EI-14	1400	760	60	Furnace	
Nickel-Copper Alloy, Cold-Worked	HE-4	400	204	180	Furnace	
Nickel-Copper Alloy, Cold-Worked	HE-14	1400	760	60	Furnace	
Copper, Electrolytic, Cold-Worked	HF-2.5	250	121	180	Furnace	
Copper, Electrolytic, Hot-Rolled	EE-12	1200	649	60	Furnace	
Aluminum	DRB	....	....	....	....	Hard Temper
Aluminum	DRA	....	....	....	....	Half Hard Temper
Aluminum	DR	....	....	....	....	Annealed
Aluminum-Manganese Alloy	DSB	....	....	....	....	Hard Temper
Aluminum-Manganese Alloy	DSA	....	....	....	....	Half Hard Temper
Aluminum-Manganese Alloy	DS	....	....	....	....	Annealed
Duralumin	GT	....	....	....	....	Tempered
Duralumin	DU	....	....	....	....	Annealed

Chemical Composition:

The chemical composition of the alloys used is given in Table I. Trade names, except those that are in universal use, have been avoided, and each material has been designated by a combination of letters.

Heat Treatment:

Details of heat treatment are given in Table II. It will be noted that, with one exception, the cold-worked nickel-copper alloys have two different heat treatments, a "low anneal" to relieve internal stress and a high-temperature anneal for complete recrystallization. The proper temperature for the "low anneal" was determined by hardness tests on a series of specimens after annealing at various temperatures. For the "low anneal" the highest temperature was selected that would cause practically no loss of strength. Internal stress measurements were not made.

Tension Tests

Results of tension tests are given in Table III. In this table "elastic limit" means the highest stress that results in no appreciable permanent deformation after removal of the load. "Proof stress" means the stress that results in a permanent deformation of 0.0001 in. per inch of length after removal of the load.

Table III.—Static Tension Test Results.

Averages and Mean Departures from Averages.

Material	Condition	Designation	Tensile Strength, lb. per sq. in.	Johnson's Limit, lb. per sq. in.	Proof Stress, lb. per sq. in.	Elastic Limit, lb. per sq. in.	Proportional Limit, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent
Nickel, Cold-Rolled	Low Anneal	EO-6	^{b}131 700 ±2 700	^{b}95 200 ±4 900	^{b}92 000 ±4 000	^{b}86 000 ±3 000	^{b}85 000 ±3 000	^{b}16.2 ±0.7	^{b}34.1 ±2.1
Nickel, Cold-Rolled	Fully Annealed	EO-14	77 600 ±300	22 000 ±1 300	21 800 ±1 900	19 300 ±2 900	19 000 ±2 700	49.3 ±2.4	75.2 ±0.8
Monel Metal, Cold-Rolled	Low Anneal	EP-8	^{b}127 200 ±1 000	^{b}95 000 ±1 000	^{b}90 000 ±1 300	^{b}84 500 ±800	^{b}82 000 ±2 000	^{b}20.6 ±0.6	^{b}57.8 ±1.5
Monel Metal, Cold-Rolled	Fully Annealed	EP-14	^{b}81 900 ±100	^{b}31 000 ±1 500	^{b}31 900 ±1 400	^{b}28 200 ±2 700	^{b}25 000 ±1 500	^{b}48.9 ±0.3	^{b}73.6 ±0.4
Nickel-Copper Alloy, Cold-Rolled	As Received	EI	^{a}85 800 ±800	^{a}56 000 ±1 000	^{a}55 800 ±300	^{a}47 500 ±2 500	^{a}38 000 ±2 000	^{a}34.5 ±2.0	^{a}60.3 ±2.3
Nickel-Copper Alloy, Cold-Rolled	Fully Annealed	EI-14	^{a}78 000	^{a}30 300 ±1 300	^{a}30 000 ±1 000	^{a}26 000 ±1 000	^{a}22 800 ±300	^{a}45.5 ±0.5	^{a}61.5 ....
Nickel-Copper Alloy, Cold-Worked	Low Anneal	HE-4	62 400 ±200	44 700 ±1 800	44 700 ±1 100	34 300 ±1 800	24 700 ±2 400	22.5 ±1.0	68.2 ±0.8
Nickel-Copper Alloy, Cold-Worked	Fully Annealed	HE-14	47 300 ±600	11 000 ±700	11 700 ±1 500	9 200 ±1 100	8 100 ±1 200	50.0 ±1.7	74.7 ±1.9
Copper, Electrolytic, Cold-Worked	Low Anneal	HF-2.5	46 500 ±200	31 000 ±700	21 200 ±1 100	12 000 ±700	9 200 ±600	14.3 ±0.6	55.8 ±2.1
Copper, Electrolytic, Hot-Rolled	Fully Annealed	EE-12	^{a}31 200	^{a} ^{d}	^{a}4 500	^{a}3 000	^{a} ^{d}	^{a}73.8 ±0.3	^{a}52.8 ±0.3
Aluminum, Hard Temper	As Received	DRB	20 500 ±200	15 300 ±400	15 600 ±300	12 800 ±900	12 700 ±200	18.0 ±0.3	64.3 ±0.7
Aluminum, Half Hard Temper	As Received	DRA	16 000 ±200	12 300 ±400	12 400 ±300	8 700 ±900	7 800 ±400	23.3 ±1.2	75.3 ±0.4
Aluminum, Annealed	As Received	DR	12 600 ±100	.... ^{d}	2 900 ±300	2 000	.... ^{d}	48.0 ±0.7	80.9 ±0.3
Aluminum-Manganese Alloy, Hard Temper	As Received	DSB	29 600 ±400	20 800 ±800	19 800 ±800	13 300 ±600	11 500 ±700	12.3 ±1.4	46.3 ±2.8
Aluminum-Manganese Alloy, Half Hard Temper	As Received	DSA	23 800 ±900	18 000 ±300	17 300 ±400	13 000 ±700	9 300 ±800	19.7 ±1.1	57.3 ±0.6
Aluminum-Manganese Alloy, Annealed	As Received	DS	16 700 ±200	2 700 ±200	3 100 ±400	2 200 ±200	1 900 ±400	44.2 ±0.9	70.3 ±1.7
Duralumin, Tempered	As Received	GT	^{c}69 100 ±900	^{c}45 300 ±1 100	^{c}45 500 ±1 100	^{c}40 700 ±900	^{c}36 800 ±600	^{c}17.8 ±1.2	^{c}22.8 ±2.2
Duralumin, Annealed	As Received	DU	^{b}33 400 ±100	^{b}19 800 ±900	^{b}20 300 ±300	^{b}17 900 ±200	^{b}15 100 ±200	^{b}17.5 ±0.3	^{b}42.7 ±0.5

[a] Average of 2 determinations.
[b] Average of 4 determinations.
[c] Average of 6 determinations.
[d] Stress-strain graph curved from origin. Unless otherwise indicated each value is average of three determinations.

PART II.—CORROSION-FATIGUE OF NICKEL, COPPER AND NICKEL-COPPER ALLOYS

General Description and Method of Test:

Stress-cycle graphs illustrating fatigue and corrosion-fatigue of nickel, copper, and nickel-copper alloys are shown in Figs. 1 to 4, inclusive. In these figures the solid line curves represent stress-cycle relationship for specimens tested in air. The broken-line curves represent stress-cycle relationship for specimens tested in either fresh or salt water. The fresh and salt water used are the same that were used in the fatigue tests on steel described in the first and second papers.(1,2) The fresh water is a carbonate water, the composition of which was given in the second paper.(2) The salt water is Severn River water having a saline content about one-third that of sea water.

The method of applying the water stream was described in the two previous papers.(1,2) During the winter months the water before reaching the specimen was artificially heated to a temperature between 60 and 70° F., which was the temperature used in the tests on steels described in the two previous papers. The stress-cycle graphs have been plotted on a semi-logarithmic scale.

Beside each stress-cycle graph there are indicated some of the strength values as determined by tension test. These are represented by the following symbols: Tensile strength, *T. S.;* Johnson's limit, *J. L.;* proof stress, *P. S.;* elastic limit, *E. L.;* proportional limit, *P. L.*

Each zig-zag line shown in these figures represents the course of a fatigue or corrosion-fatigue test in which the stress was raised at intervals until the specimen failed. The lower end of the zig-zag line represents the original stress and the number of cycles endured at this stress. The course of the line represents the stress increases and the cycle intervals at which these increases were made.

Nickel:

Fatigue and corrosion-fatigue graphs for nickel are shown in Fig. 1. The series of graphs on the right of Fig. 1 represents nickel that had been cold rolled and then given a "low anneal" to relieve internal stress with practically no reduction in strength. The series of graphs on the left represents fully annealed material. Each series of graphs has its own scale of abscissas which is indicated by figures along the graph. For both series of graphs the scale of ordinates is the same.

Results of ordinary endurance tests in air are indicated by the small circles, and the stress-cycle relationship is indicated by the curved solid lines. The fatigue graphs are typical graphs for such material. They consist of curves descending toward a horizontal asymptote, the ordinate of which represents the "endurance limit." When extended upward sufficiently there is the usual reversal of curvature which has been described in previous papers.(1,2)

Results of corrosion-fatigue tests in fresh water are represented by the small open triangles, and by the accompanying curved broken

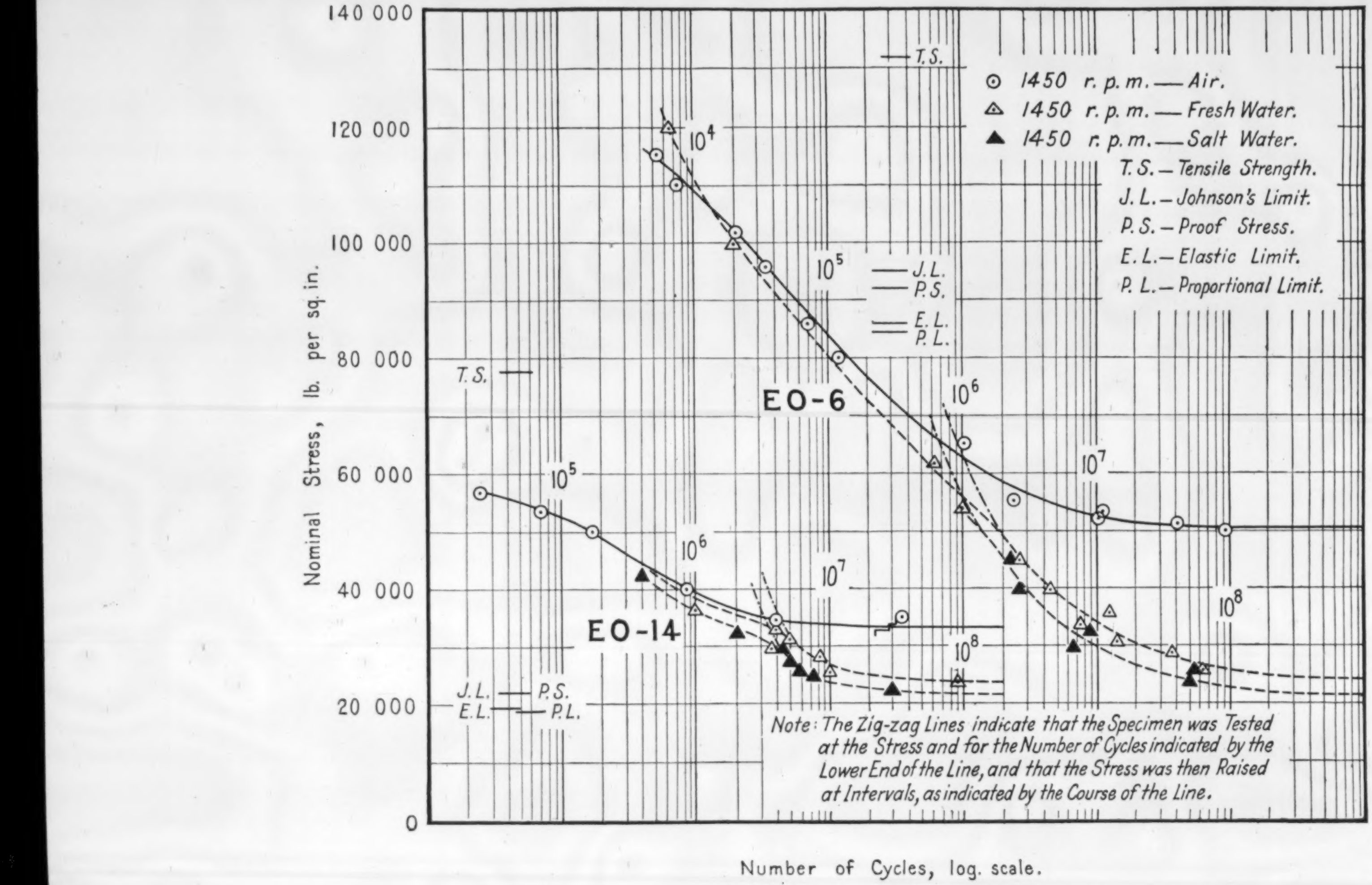


FIG. 1.—Fatigue and Corrosion-Fatigue of Nickel.

lines. Results of corrosion-fatigue tests in salt water are represented by the small solid triangles and by the accompanying broken lines.

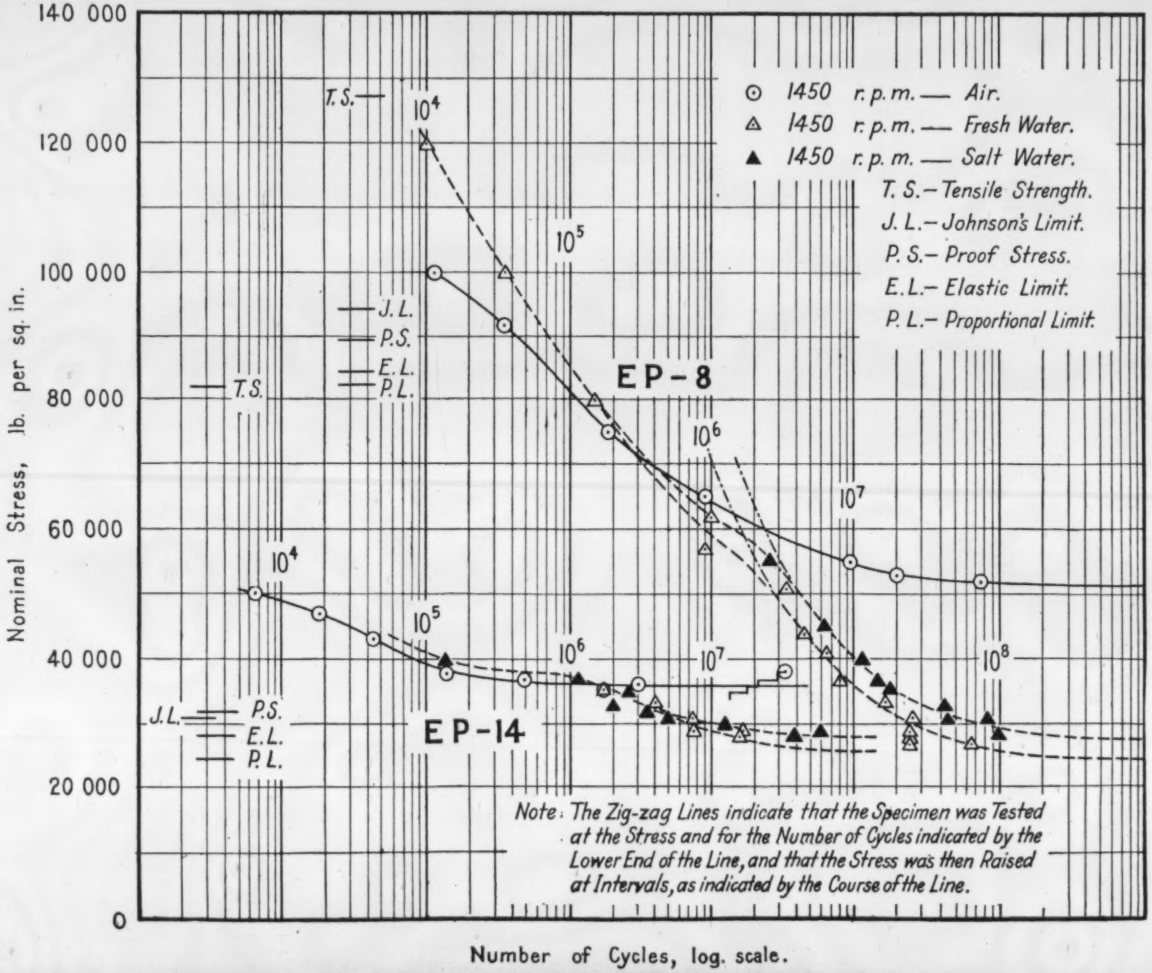
It will be noted that each of these corrosion-fatigue graphs for nickel, as do the corrosion-fatigue graphs for steel shown in the second paper,(2) descends toward a horizontal asymptote, the "corrosion-fatigue limit." The most striking phenomenon is that the corrosion-fatigue limits for the strain-hardened and for the fully-annealed nickel are practically the same. Although cold working increases the endurance limit about in proportion to the increase in tensile strength, it has practically no effect on the corrosion-fatigue limit of nickel. This phenomenon, however, was not entirely unexpected in view of the fact that for steel, as shown in the previous papers(1)(2), the corrosion-fatigue limit is little influenced by composition or heat treatment, except as the composition or heat treatment influences resistance to corrosion.

The form of the corrosion-fatigue graphs is of considerable interest. At stresses well above the endurance limit each corrosion-fatigue graph has downward convexity and runs nearly parallel to the corresponding fatigue graph. At some point in its descent, however, the corrosion-fatigue graph reverses its curvature and begins to descend rapidly below the fatigue graph. With further descent there is a second reversal of curvature and the stress-cycle line now approaches a horizontal asymptote. These stress-cycle lines, therefore, are made up of three distinct curves. Other corrosion-fatigue graphs to be shown later are made up of four distinct curves. It is worthy of note that the point of lowest reversal of curvature is near the endurance limit of the material.

For nickel the abscissa at which the corrosion-fatigue graph begins to descend rapidly below the corresponding fatigue graph is greater than for steels, except possibly for some corrosion-resistant steels. Whereas for carbon steels, the corrosion-fatigue and fatigue graphs begin to separate at a hundred thousand cycles or less, for strain-hardened nickel the point of separation is at nearly a million cycles, and for fully annealed nickel the corresponding point is at about four million cycles.

Monel Metal:

Fatigue and corrosion-fatigue graphs for monel metal are shown in Fig. 2. As in Fig. 1, the series of graphs on the right of Fig. 2 represents strain-hardened material and the series on the left represents fully annealed material. A comparison of the corrosion-fatigue graphs shows that the corrosion-fatigue limits for strain-hardened and for fully annealed monel metal are practically the same. For monel



Note: The Zig-zag Lines indicate that the Specimen was Tested at the Stress and for the Number of Cycles indicated by the Lower End of the Line, and that the Stress was then Raised at Intervals, as indicated by the Course of the Line.

FIG. 2.—Fatigue and Corrosion-Fatigue of Monel Metal.

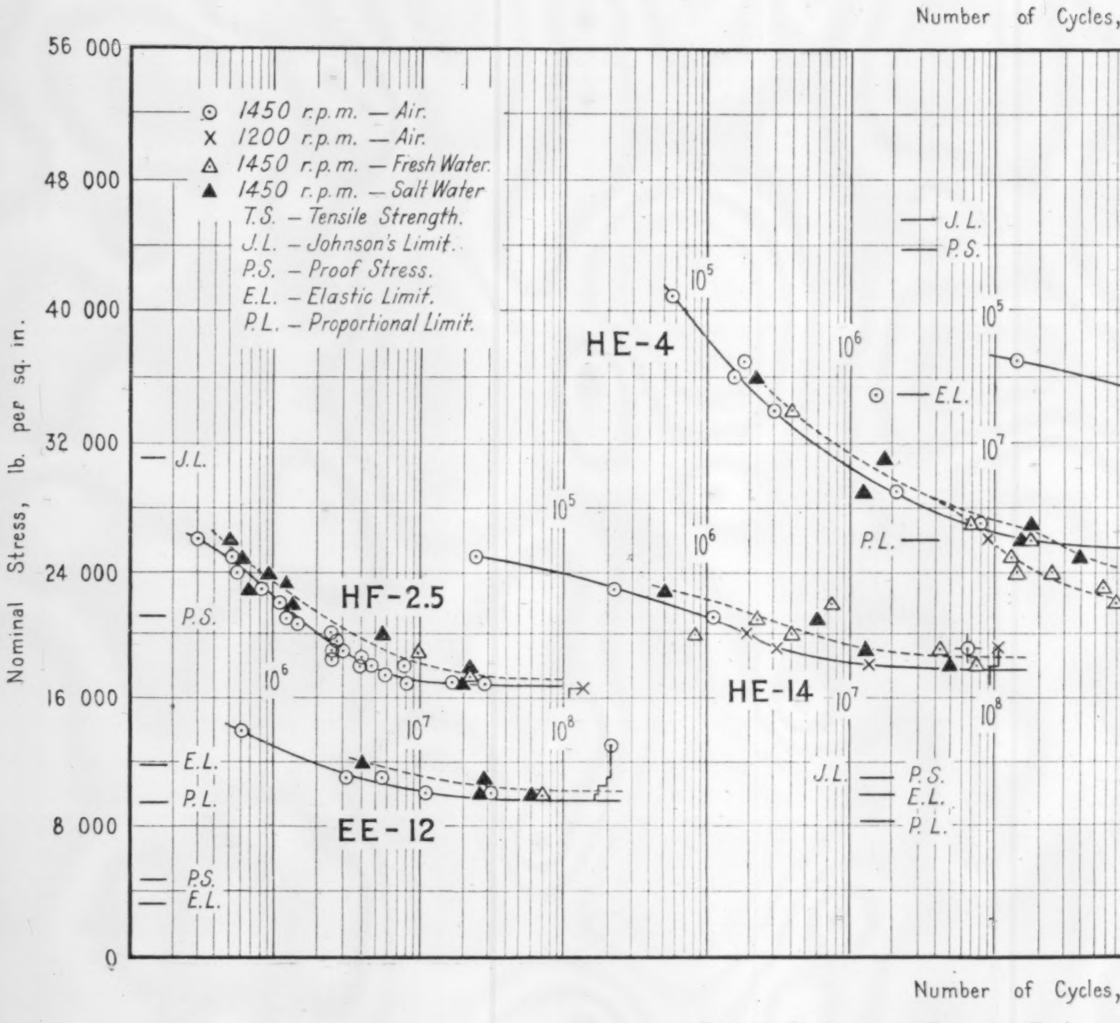


Fig. 3.—Fatigue and Corrosion-Fatigue c

PLATE I.
PROC. AM. SOC. TESTING MATS.
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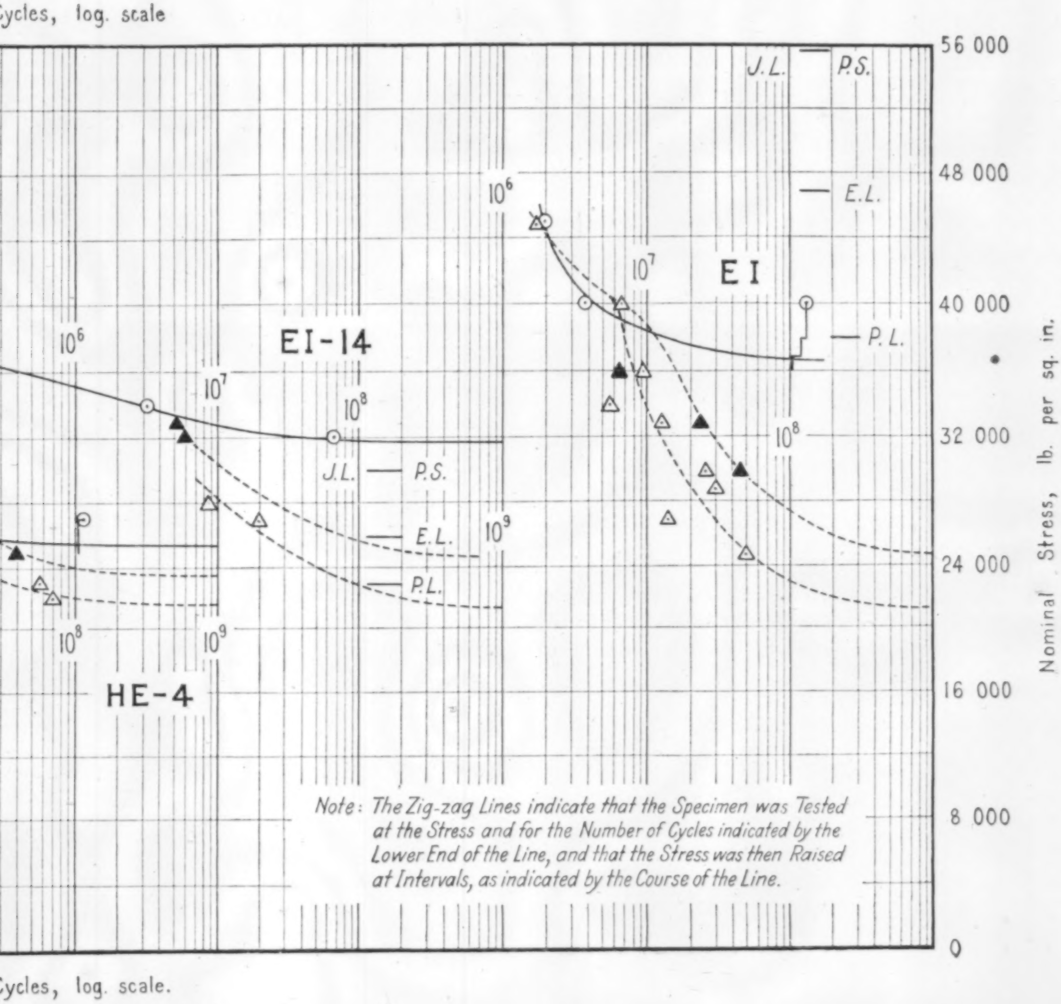
MCADAM ON CORROSION-FATIGUE OF NON-FERROUS METALS.



Note: The Zig-zag Lines indicate that the Specimen was Tested at the Stress and for the Number of Cycles indicated by the Lower End of the Line, and that the Stress was then Raised at Intervals, as indicated by the Course of the Line.

igue of Some Nickel-Copper Alloys.

metal as for nickel, therefore, cold-working has practically no effect on the corrosion-fatigue limit. A striking difference between Figs. 1 and 2, however, is in the relative positions of the corrosion-fatigue graphs for fresh and salt water. For nickel, the fresh-water corrosion-fatigue graph is above the salt-water corrosion-fatigue graph; for monel metal the fresh-water graph is the lower. It is worthy of note that the fresh-water corrosion-fatigue limits for nickel and for monel metal are nearly the same.

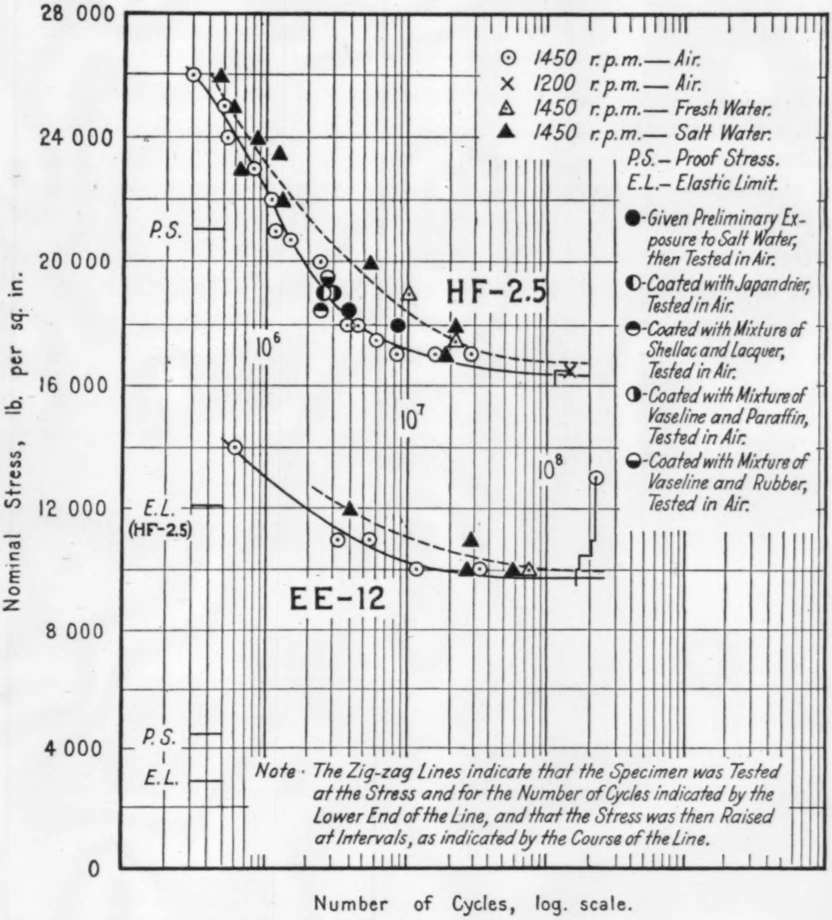


Fig. 4.—Fatigue and Corrosion-Fatigue of Copper.

For either material, however, there is much less difference between the corrosion-fatigue limits in fresh and salt water than for the corrosion-resistant steels illustrated in the second paper.(2) As shown in that paper, for corrosion-resistant steels the salt-water corrosion-fatigue limit is much lower than the fresh water corrosion-fatigue limit.

The form of the corrosion-fatigue graph for monel metal is similar to the form of the corrosion-fatigue graph for nickel. As shown in Fig. 2, each corrosion-fatigue line is made up of three curves, two curves with downward convexity connected by a short curve with upward convexity. At the higher stresses, however, the corrosion-fatigue graphs for monel metal are above the ordinary fatigue graphs; in this respect they differ from the corresponding graphs for nickel. As will be shown later, the relative positions of the upper parts of the fatigue and corrosion-fatigue graphs depend on the opposing chemical and thermal effects of the water stream.

The point at which the corrosion-fatigue graph for monel metal begins to descend rapidly below the corresponding fatigue graph is at nearly a million cycles for the strain-hardened material and at more than two million cycles for the fully-annealed material. This delayed descent accounts for the fact that corrosion-fatigue of monel metal was not observed in a previous investigation,(1) in which a stream of fresh water was used for its cooling effect. The point of lowest reversal of curvature of the corrosion-fatigue graph for monel metal as for nickel is near the endurance limit. Possible reasons for this will be discussed later.

The significance of the dot-and-dash lines in Figs. 1 and 2 will be discussed later.

48:48 Nickel-Copper Alloy:

In Fig. 3 are shown three pairs of graphs arranged from left to right in order of increasing nickel content of the material represented. The two series on the left represent electrolytic copper; the two middle series represent an alloy containing about 21 per cent of nickel; and the two series on the right represent an alloy containing equal proportions of nickel and copper, with proportions of iron and manganese somewhat similar to those in monel metal. The mode of representation of fatigue and corrosion-fatigue graphs in Fig. 3 is the same as in Figs. 1 and 2.

For the 48:48 nickel-copper alloy, materials EI and EI-14, the corrosion-fatigue graphs for salt water are above those for fresh water. In this respect the alloy resembles monel metal. It will be noted that the rapid descent of the corrosion-fatigue graphs below the fatigue graphs does not begin before nearly ten million cycles.

As this delayed descent of the corrosion-fatigue graphs gave the impression that the corrosion-fatigue limits would be not far below the corresponding endurance limits, the number of specimens reserved was insufficient to determine definitely the corrosion-fatigue limits. Apparently, however, for material EI the corrosion-fatigue limits are no higher than for monel metal and for the 21:78 nickel-copper alloy to be described below. For this reason the corrosion-fatigue graphs for this intermediate alloy have been drawn on the assumption that these limits are practically the same as for monel metal. For the same reason the corrosion-fatigue graphs for the fully annealed material EI-14 have been drawn so as to approach the same corrosion-fatigue limits as those of the strain-hardened material EI.

21:78 Nickel-Copper Alloy:

As shown in Fig. 3 the fatigue graphs for the strain-hardened material HE-4 and for the fully annealed material HE-14 are of the usual form. Each consists of a curve descending toward a horizontal asymptote and extending upward into a reversed curve.

For material HE-4 the corrosion-fatigue graphs in their upper regions run nearly parallel to the fatigue graph. At about ten million cycles the fresh water corrosion-fatigue graph begins its descent below the fatigue graph. It descends toward a corrosion-fatigue limit of about 22,000 lb. per sq. in. The salt water corrosion-fatigue graph does not begin its descent before about twenty million cycles, and the salt water corrosion-fatigue limit is evidently very slightly below the endurance limit. As previously noted, the corrosion-fatigue limits for the strain-hardened alloy are nearly the same as for monel metal.

The endurance limit of the fully annealed material HE-14 is well below the corrosion-fatigue limits of the strain-hardened material HE-4. In this respect the 21:78 nickel-copper alloy differs from the nickel-copper alloys previously described. It is not surprising, therefore, to find that for material HE-14 the graph representing results of tests in fresh and salt water is above the ordinary fatigue graph. This elevation of the stress-cycle graph is probably due not to the chemical action of the water but to the cooling effect. This effect has been discussed in a previous paper.(1) In the upper part of the graph for material HE-14 there is considerable "scatter" of the individual results. Apparently, however, there is practically no difference between the effects of fresh and salt water on this material. This might have been expected, as the effect of the water is chiefly thermal.

Evidently, therefore, true corrosion-fatigue phenomena have not been encountered with material HE-14 because of the physical limitations of this material. The intrinsic corrosion-fatigue limit for material HE-14 is above the endurance limit. Only after a suitable strengthening process such as cold working can the intrinsic corrosion-fatigue limit for this alloy be encountered.

Copper:

Results of fatigue tests of copper in air, fresh water and salt water are shown at the left end of Fig. 3, and on a larger scale in Fig. 4. The fatigue graph for the strain-hardened copper, HF-2.5, tested in air is of the typical form, a curve with horizontal asymptote and reversing its curvatures with upward extension. The hot-rolled copper, EE-12, is evidently in a fully softened condition. The fatigue graph has not been extended upward far enough to encounter reversal of curvature.

For both the strain-hardened and hot-rolled copper the stress-cycle lines representing specimens tested in water are above the corresponding lines representing specimens tested in air. This absence of true corrosion-fatigue phenomena is not due to lack of chemical action, for the surfaces of the specimens tested in water become heavily coated with films of corrosion products. It was thought possible that a protective action of these films might account for the elevation of the stress-cycle line due to action of the water stream. Such elevation of the graph due to protective action of a film could occur only if the ordinary fatigue graph is too low owing to atmospheric oxidation of the copper. This hardly seemed possible, as all specimens tested in air at the Naval Experiment Station had been kept covered with a film of mineral oil.

Nevertheless, to test this hypothesis some specimens were given a preliminary coating due to action of salt water without accompanying stress. They were then subjected to a fatigue test in air. The results are represented in Fig. 4 by the small solid circles. Although one of these circles is beyond the average position of the ordinary fatigue graph the other circle is almost exactly on the fatigue graph. Neither specimen ran as long as the corresponding water-cooled specimens.

To test still further, however, the possibility that the ordinary fatigue graph for copper may be too low, owing to atmospheric corrosion, specimens were given protective coatings and tested in air. Some of these protective coatings were suggested, in a private communication, by H. F. Moore, who had tried them in some fatigue tests on steel. The coatings used were: Mixture of vaseline and rubber, mixture of vaseline and paraffin, Japan drier, and mixture of shellac and lacquer. As shown in Fig. 4, all these specimens broke at or near the ordinary fatigue graph. This seems to indicate that the ordinary fatigue graph has not been influenced by atmospheric corrosion.

If the solid-line curves in Fig. 4 represent true fatigue graphs, the elevation of the stress-cycle graph due to action of the water stream is due not to its chemical action but to its cooling effect. That water cooling of copper specimens would have such a conspicuous effect was not anticipated. Such effects of water cooling have been described in a previous paper,(1) but those effects were obtained with monel metal and other alloys of low thermal conductivity.

It will be noted that the distance between the graph for water-cooled specimens and the corresponding fatigue graph is greatest at the region of most rapid curvature of the fatigue graph. As the two graphs are extended to the right, they approach each other. The asymptote of the upper graph, however, is probably slightly above the asymptote of the ordinary fatigue graph. Reasons for this conclusion have been given in the previous paper.(1)

Evidently, therefore, for both the strain-hardened and hot-rolled copper the intrinsic corrosion-fatigue limit for fresh or salt water is above the endurance limit. In this respect copper is similar to the fully annealed 21:78 nickel-copper alloy previously discussed. This relationship is illustrated in Fig. 3. Starting at the left of the figure the endurance limit increases from that of hot-rolled copper to that of strain-hardened material HE-4. For the first three graphs the endurance limit is below the intrinsic corrosion-fatigue limit. Only when the fourth series of graphs is reached does the endurance limit surpass the corrosion-fatigue limit.

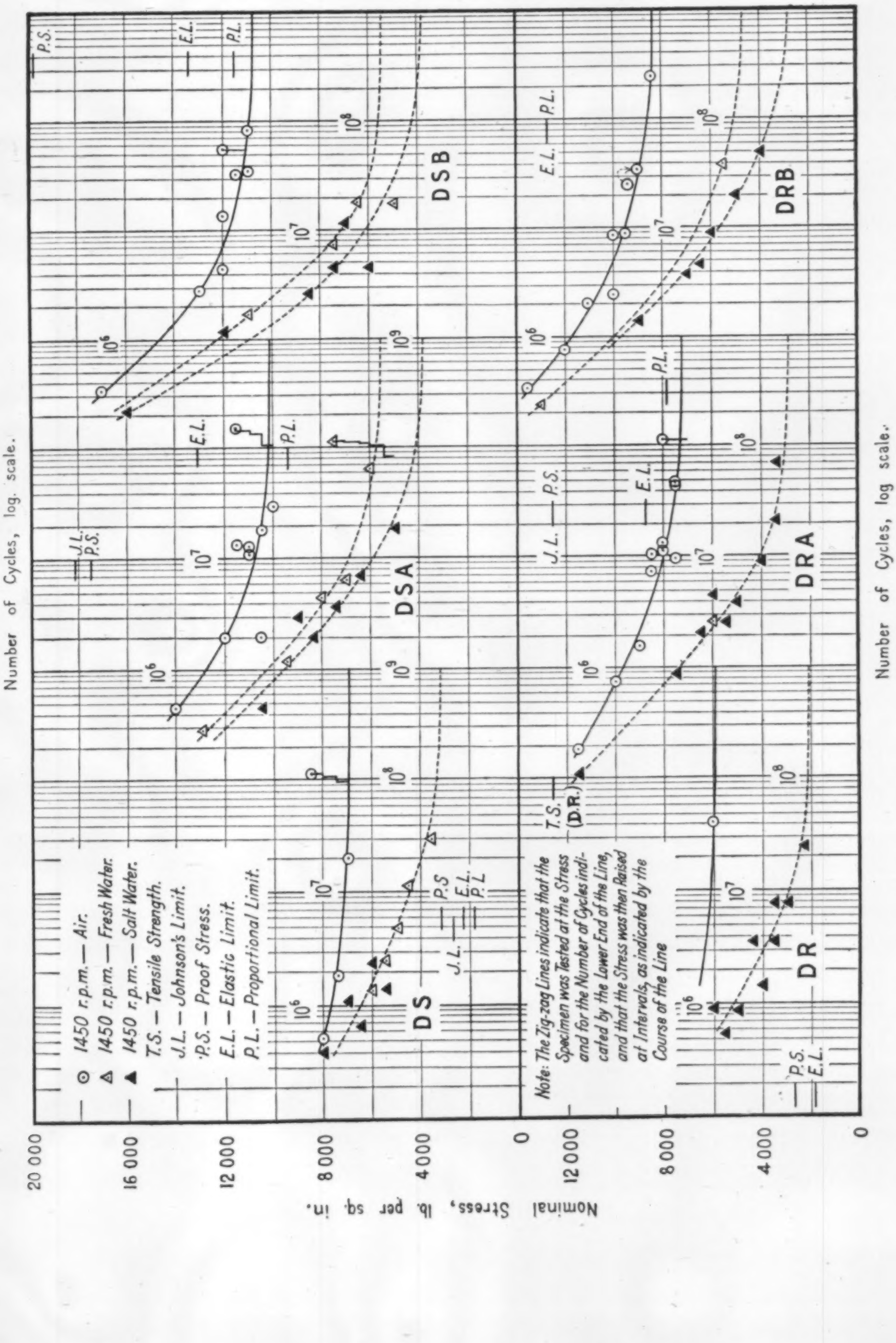
Relationship Between Chemical Composition and Corrosion-Fatigue Limits of Nickel-Copper Alloys:

A rather surprising relationship seems to be brought out by comparison of the corrosion-fatigue limits indicated in Figs. 1 to 3 inclusive. The corrosion-fatigue limits of monel metal, of the 21:78 alloy, and probably of the 48:48 alloy are practically the same. Moreover the fresh-water corrosion-fatigue limits for these alloys are practically the same as for nickel. Subject to relatively slight variations due to influence of oxides, non-metallic inclusions, and other metallic elements present in small quantities, it seems probable that the intrinsic corrosion-fatigue limits for nickel-copper alloys are practically the same, at least for alloys having between 21 and 99 per cent of nickel. To determine the full composition range for this relative constancy of the corrosion-fatigue limit experiments will be necessary with alloys having less than 21 per cent of nickel. It seems not improbable, however, that the composition range of constancy of the intrinsic corrosion-fatigue limits extends from 99 per cent of nickel nearly to pure copper. This idea is suggested by consideration of the electrolytic potentials of the nickel-copper alloys.

To account for the observed wide composition range of constancy of the corrosion-fatigue limit, two ideas suggest themselves. A possible reason for the constancy would be the presence on the intercrystallin boundaries of small quantities of a second phase having nearly constant composition for the entire composition range of nickel-copper alloys. Such a constant intercrystallin phase conceivably might cause intercrystallin failure under corrosion-fatigue at about the same stress range for the entire series of alloys. Purely intercrystallin failure, however, for these alloys under corrosion-fatigue has not yet been observed.

The other idea that is suggested by the observed constancy of the corrosion-fatigue limits for a wide range of composition is that the corrosion-fatigue limits for the nickel-copper series of alloys may depend chiefly on the electrolytic potential of the alloys. In a paper by N. T. Gordon and D. P. Smith(8) it was shown that the electrolytic potential of the nickel-copper alloys in a normal solution of their mixed sulfates varies from +0.585 to +0.563 volt over a range of composition from 5.3 to 93.2 per cent of nickel. Some preliminary experiments at the Naval Experiment Station indicate that the electrolytic potential varies only slightly throughout the entire composition range from pure copper to 99 per cent of nickel. For electrolytically deposited nickel the potential obtained was less positive but was still considerably higher than the very slightly positive values for nickel given in handbooks.

The electrolytic potential, therefore, evidently varies only slightly over the range of commercial nickel-copper alloys. Whether this relative constancy of the electrolytic potential has any theoretical connection with the observed relative constancy of the intrinsic corrosion-fatigue limit or is merely a coincidence must be decided later. It should be pointed out that the modulus of elasticity of the nickel-copper alloys decreases from about 28,000,000 for nickel to about 18,000,000 lb. per sq. in. for copper. If modulus of elasticity has any effect on the corrosion-fatigue limit, its tendency, if acting



Note: The Zig-zag Lines indicate that the Specimen was Tested at the Stress and for the Number of Cycles indicated by the Lower End of the Line, and that the Stress was then Raised at Intervals, as indicated by the Course of the Line

FIG. 5.—Fatigue and Corrosion-Fatigue of Aluminum and Aluminum-Manganese Alloys.

alone, would be to cause a uniform drop in the intrinsic corrosion-fatigue limit from nickel to copper. The tendency of the slight range of electrolytic potential, if taken alone, might cause a slight rise in the intrinsic corrosion-fatigue limit from nickel to copper.

PART III.—CORROSION-FATIGUE OF ALUMINUM AND ALUMINUM ALLOYS

Aluminum:

Fatigue and corrosion-fatigue graphs for aluminum are shown in the lower half of Fig. 5. The material was tested in three degrees of hardness, which have been designated by the Aluminum Co. of America as "soft," "half hard," and "hard." The graphs are arranged from left to right in order of increasing hardness.

The fatigue graphs for aluminum are asymptotic curves. They have not been extended upward far anough to encounter reversal of curvature. The graph for soft aluminum is unusually flat. For the soft aluminum the usual type of test specimen could not be used. The endurance limit is so far above the elastic limit that the heat evolved in an endurance test weakens the specimen enough to cause immediate bending until the specimen is useless. For this reason a thicker specimen with greater ratio of heat conductivity to heat evolution was designed. With this specimen endurance tests could be made. Even thus, however, the fatigue graph is practically horizontal.

The increase of the endurance limit of aluminum by cold working is slightly less than in proportion to the increase in tensile strength. The rate of increase of the endurance limit seems to decrease with increase in hardness.

The salt-water corrosion-fatigue graphs have been extended far enough to show that for aluminum the corrosion-fatigue limit is far below the endurance limit. Sufficient specimens were not available to determine the corrosion-fatigue limits with accuracy. If, however, the graphs for aluminum be studied in connection with the graphs for the aluminum-manganese alloy in the upper half of Fig. 5, the evidence seems to indicate that for half-hard and for hard aluminum the corrosion-fatigue limits are about the same, and that for the soft alloy the corrosion-fatigue limit is slightly lower. For soft aluminum the corrosion-fatigue limit is apparently close to the elastic limit.

Aluminum-Manganese Alloy:

Fatigue and corrosion-fatigue graphs for the aluminum-manganese alloy are shown in the upper half of Fig. 5. The graphs for soft, half hard, and hard material are arranged in order from left to right.

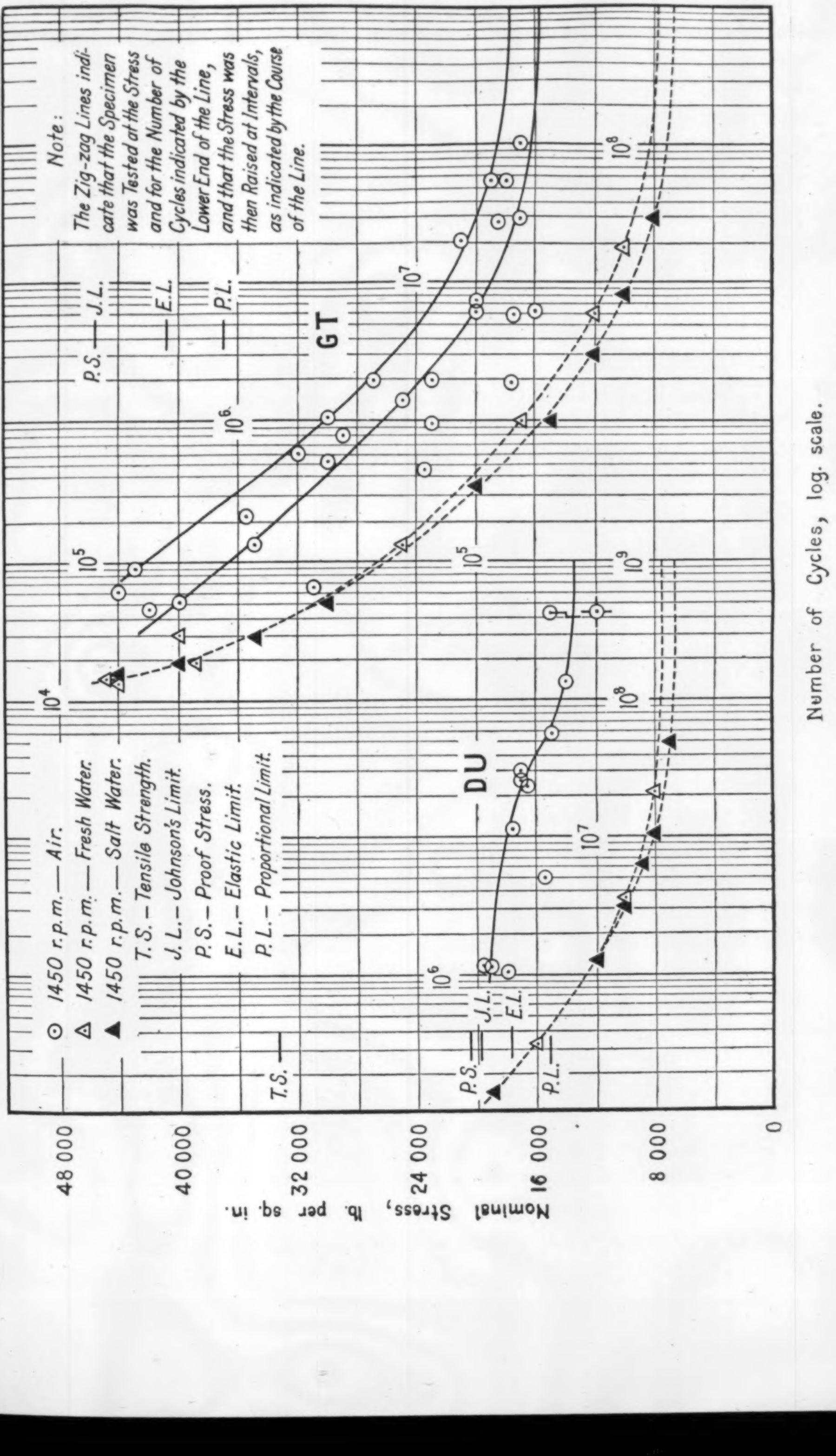


FIG. 6.—Fatigue and Corrosion-Fatigue of Duralumin.

The fatigue graphs for this aluminum-manganese alloy are asymptotic curves. The graphs for half-hard and for hard material have not been extended upward far enough to encounter reversal of curvature. The graph for the soft alloy is so nearly horizontal that it is difficult to note whether or not there is reversal of curvature. The endurance limits of the half-hard and of the soft alloy are about in proportion to their tensile strength. With further increase in hardness, however, the increase in the endurance limit is less than in proportion to the increase in tensile strength.

Sufficient specimens were not available for the exact determination of corrosion-fatigue limits. The evidence seems to indicate, however, that the fresh-water corrosion-fatigue limit is about the same for half-hard and for hard material. For soft material the corrosion-fatigue limit appears to be decidedly lower. For the aluminum-manganese alloy, as for aluminum, the corrosion-fatigue limit of soft material is only slightly, if at all, above the elastic limit.

Duralumin:

Fatigue and corrosion-fatigue graphs for duralumin are shown in Fig. 6. The series on the right represents duralumin of unusually hard temper. The series on the left represents fully-annealed duralumin.

For the tempered duralumin the results of ordinary fatigue tests were unusually erratic. The portion of the field included between the upper and lower solid line curves for Material GT represents about the usual scatter for duralumin. This has been illustrated in a previous paper.(9) This wide scatter is not due to variation in the surface finish of the specimens and is probably not due to atmospheric corrosion. These specimens during tests were covered with a film of mineral oil. Some specimens had been given the anodic oxidation treatment that has been recommended for inhibition of corrosion. This caused no dimunition of the scatter.

Results of corrosion-fatigue tests for this material, however, are unusually consistent, as shown by the graphs representing fresh and salt-water corrosion-fatigue. The corrosion-fatigue graphs throughout a wide range of stress are much below the fatigue graphs. A few of the individual results of fatigue tests, however, are not far beyond the corrosion-fatigue graphs. The rapidity with which corrosion-fatigue of duralumin manifests itself is strikingly illustrated in the figure. For a range of stress from 44,000 to 26,000 lb. per sq. in. the range of abscissas of the corrosion-fatigue graph corresponds to a time range from ten minutes to one and one-quarter hours.

The fatigue graph for annealed duralumin shows a decided reversal of curvature with upward extension. The endurance limit of this material is not far below the endurance limit of the tempered material, although the tensile strength of the former is less than half that of the latter. For the annealed duralumin, as for the tempered duralumin, the corrosion-fatigue results are more consistent than are the fatigue results. This could not easily be accounted for by a mechanical stress-concentration theory.

The corrosion-fatigue limits for the tempered and for the annealed duralumin are practically the same. Apparently, heat treatment of this material has very little influence on the corrosion-fatigue limit. In this respect duralumin is similar to steel.

The fact that the corrosion-fatigue limits for duralumin are so low, emphasizes the importance of protective coatings for this material. Experiments are now under way to determine the effect of various coatings on the corrosion-fatigue limit.

PART IV.—GENERAL DISCUSSION OF CORROSION-FATIGUE OF METALS

Complete theoretical discussion must be deferred until additional series of binary alloys have been investigated and presented in later papers. Nevertheless, it seems advisable to discuss tentatively some hypotheses suggested by facts presented in two previous papers (1,2) and in the present paper. It should be understood that any theoretical discussion presented now is subject to revision as additional information becomes available.

Intrinsic Corrosion-Fatigue Limits:

The fact that for carbon steels and all alloy steels except corrosion-resistant steels the corrosion-fatigue limit varies surprisingly little with composition and heat-treatment, and the fact that for each increase in corrosion resistance by addition of elements such as chromium and nickel there is an increase in the corrosion-fatigue limit, first suggested vaguely the idea that for each metal and alloy there may be an intrinsic corrosion-fatigue limit depending chiefly on the corrosive agent and on the corrosion-resistance of the material. This hypothesis received support when it was found that for nickel-copper alloys the corrosion-fatigue limit is practically unaffected by cold working. It received additional confirmation when it was found that for duralumin the corrosion-fatigue limit is practically unaffected by heat treatment. And there are yet other alloys each showing constancy of the corrosion-fatigue limit under varying conditions of cold working and heat treatment. These will be presented in a later paper. These examples seem too numerous to be ascribed to coincidence. Aluminum and the aluminum-manganese alloy, as represented in Fig. 5, are the only alloys yet encountered for which the corrosion-fatigue limit is considerably affected by cold-working. Even for these apparent exceptions, however, the increase in corrosion-fatigue limit by cold working is probably confined to the slight amount of cold working necessary to raise the elastic limit until it no longer restricts the corrosion-fatigue limit. Beyond this point cold working probably has practically no effect on the corrosion-fatigue limit.

It was pointed out in detailed description of Fig. 5, that for the annealed aluminum and aluminum-manganese alloy the corrosion-fatigue limits are not far above the tensile elastic limit. It is possible that the lower corrosion-fatigue limit for the soft material is due to restriction of the corrosion-fatigue limit by an elastic limit. No such restriction, however, is apparent for the annealed 21:78 nickel-copper alloy, material HE-14 of Fig. 3; for that alloy the corrosion-fatigue limit is restricted only by the endurance limit, which is far above the tensile elastic limit. It is difficult to see why the elastic limit should restrict the corrosion-fatigue limit for one alloy but not for another. The restriction limit, however, may correspond not to the tensile elastic limit but to an elastic limit or a slight amount of plastic deformation in bending. Possibly the intrinsic corrosion-fatigue limit may really be a deformation limit rather than a stress limit. This subject needs further investigation before definite conclusions are drawn.

Table IV.—Intrinsic Corrosion-Fatigue Limits of Alloys.

Material	Intrinsic Corrosion-Fatigue Limit, lb. per sq. in.	
	Fresh Water	Salt Water
Nickel	21 000 - 24 000	20 000 - 22 000
Monel Metal	21 000 - 24 000	27 000 - 29 000
48:48 Nickel-Copper Alloy	21 000 - 24 000	23 000 - 25 000
21:78 Nickel-Copper Alloy	21 000 - 24 000	23 000 - 25 000
Aluminum	4 000 - 5 000	2 500 - 3 500
Aluminum-Manganese Alloy	5 000 - 6 000	3 500 - 4 500
Duralumin	7 000 - 9 000	6 000 - 8 000

Even if, as seems incredible, all the examples of constancy of the corrosion-fatigue limit are merely coincidences due to the accidental balancing of two opposing influences, nevertheless, for all practical purposes it may be considered true that these alloys have intrinsic corrosion-fatigue limits, which are realized in practice only if there is no restriction due to elastic limit or endurance limit. In Table IV are listed the approximate intrinsic corrosion-fatigue limits of some alloys. Each range of values for the intrinsic corrosion-fatigue limit, given in this table, is subject to revision as other samples of the same alloy are investigated.

If the various single alloys each showing constancy of the corrosion-fatigue limit under cold working be considered separately it would seem possible to explain this constancy by surmising that corrosion-fatigue cracks start in the amorphous metal of the grain boundaries and of the slip lines caused by cold working. It does not seem incredible that under such conditions the smallest stress range necessary to start cracks in cold-worked or annealed material would be practically the same. If this hypothesis be true, one would not expect to find intercrystallin failures of cold-worked material. The fact that even for fully-annealed material no intercrystallin failures have been found does not disprove this hypothesis. The cracks might start at intercrystallin boundaries, but the extension of such cracks between two such starting points might be transcrystallin due to stress concentration.

While it may be true that corrosion-fatigue cracks start in the intercrystallin boundaries or in the "slip lines," there are other important facts of corrosion-fatigue that are not explained by this hypothesis alone. The fact that for the entire series of nickel-copper alloys, at least between 21 and 99 per cent of nickel, the intrinsic corrosion-fatigue limit is practically independent of the proportions of the two component elements, suggests another explanation, which has already been discussed briefly in this paper. It seems possible that to explain the intrinsic corrosion-fatigue limit attention must be directed to the space lattice and to inter-atomic attraction. If this be done one may imagine the corrosion-fatigue limit as depending on the ease of removal of atoms from the space lattice under the combined influence of electrolytic solution pressure and cyclic strain of the space lattice. The solution pressure for ordinary corrosion and probably for corrosion-fatigue is greatly modified by the protective action of surface films. For this reason it should not be assumed that it will be possible to predict corrosion-fatigue limits for one series of alloys from the corrosion-fatigue limits of another series by comparison of initial electrolytic potentials. For a single set of binary alloys, that forms surface films of about the same character throughout the entire range of composition, it may be possible to observe, as is apparent in the case of the nickel-copper alloys, a relationship between the corrosion-fatigue limit and electrolytic solution pressure.

Whether the hypothesis of relationship between corrosion-fatigue limit and electrolytic solution pressure will be proved or disproved by continued investigation is uncertain. Meanwhile, it will do no harm to consider it as a possibility.

Secondary Influences Affecting the Corrosion-Fatigue Limit:

Constancy of the corrosion-fatigue limit of an alloy with varying hardness through heat treatment or cold working is probably not due to any nullifying influence of varying notch toughness. Nevertheless, it seems possible that notch toughness may have a secondary influence on the corrosion-fatigue limit. Owing to surface irregularities and inclusions in the specimen the actual stress is always more or less greater than the nominal stress. Such stress concentration is greater the harder the material. This may account for the fact, presented in the first two papers (1,2) and confirmed by more recent experiments, that the apparent corrosion-fatigue limit for very hard steels is lower than for the same steels in the annealed condition. If test specimens of such material were free from surface imperfections and from inclusions, the apparent corrosion-fatigue limit for very hard and for soft conditions of the same steel might be more nearly the same.

There is some evidence that internal stress may lower the apparent corrosion-fatigue limit. It has been shown that internal stress may lower the endurance limit. It would, therefore, not be surprising to find that it lowers the corrosion-fatigue limit.

Relationship Between Fatigue and Corrosion-Fatigue:

If corrosion-fatigue be considered merely as fatigue in which the actual stress is higher than the nominal on account of stress concentration, it might be expected that all corrosion-fatigue graphs would be simple asymptotic curves, such as those for aluminum alloys shown in Figs. 5 and 6. In these graphs there is no reversal of curvature except possibly at exceptionally high stresses. On a hypothesis of mechanical stress concentration, however, it is difficult to account for complex corrosion-fatigue graphs such as those for nickel-copper alloys shown in Figs. 1 to 3, inclusive. Since such graphs were observed for nickel-copper alloys they have been encountered for a number of other alloys. Moreover, a re-examination of the corrosion-fatigue graphs for steels presented in the first two papers (1,2) seems to show that some of those graphs would have been better represented by complex curves than by the simple curves that were drawn.

On the stress-concentration hypothesis it is difficult to understand why a corrosion-fatigue graph in its downward course should first run nearly parallel to and slightly below the corresponding fatigue graph, then after apparently approaching a horizontal asymptote, abruptly reverse its curvature, descend rapidly below the fatigue graph and again approach a horizontal asymptote. A possible explanation of this complexity of such graphs is suggested by a study of the inter-relationship between the fatigue and corrosion-fatigue graphs. It will be noted that if the corrosion-fatigue graph crosses or draws near the fatigue graph the former is always of complex form. If the corrosion-fatigue graph, however, rises well to the left of the steep portion of the fatigue graph, the former is always of simple form. One receives the impression that the fatigue graph, on account of its nearness, distorts the corrosion-fatigue graph.

In studying the influence of the fatigue graph on the corrosion-fatigue graph it is illuminating to follow the course of the corrosion-fatigue graph upward rather than downward. If the course of this graph for nickel and for monel metal, Figs. 1 and 2, be thus followed, it is evident that the course of each of these graphs, if undisturbed by the influence of the fatigue graph, would cross the fatigue graph and follow the dot-and-dash line in the figure. Instead of this, however, each of these corrosion-fatigue graphs at about the endurance limit turns abruptly to the left and thereafter follows more or less closely the course of the fatigue graph. Moreover the upward course of the fatigue graph itself is to the left of what the upward course of the corrosion-fatigue graph would have been if undisturbed. This means that a specimen of nickel or monel metal tested at a stress above the endurance limit will fail sooner if tested in air than if it could be tested at the same stress under uncomplicated corrosion-fatigue.

The complicated corrosion-fatigue graph, therefore, is really made up of two graphs, a lower pure corrosion-fatigue graph and an upper graph whose course is influenced largely by some factor or factors of ordinary fatigue. The first impression given by this interrelationship is that the mechanism of corrosion-fatigue is entirely different from that of fatigue. Such a difference in mechanism might exist if corrosion-fatigue is intercrystallin, whereas, fatigue is transcrystallin. Assumption that corrosion-fatigue is intercrystallin, however, does not appear necessary at present. Another possible hypothesis is that at stresses above the endurance limit some factor is present that is not present in pure corrosion fatigue.

In a previous paper,(1) thermal influences on the form of the stress-cycle graph were discussed. An idea gained from that investigation is that in the minute regions of maximum heat evolution during a fatigue test the temperature may be considerably higher than the average temperature of the specimen. It seems possible that such elevation of temperature in minute regions may be a large factor in the above discussed influence of the fatigue graph on the form of the corrosion-fatigue graph.

A study of Figs. 1 to 3 will show that for the fully annealed alloys the influence of the fatigue graph on the form of the corrosion-fatigue graph is greater than for the strain-hardened alloys. This means that the disturbing influence of the fatigue graph is greatest when the heat evolution is greatest. It will be noticed also that for alloys of low thermal conductivity the disturbing influence of the fatigue graph is greater than for alloys of high thermal conductivity. This means that the disturbing influence of the fatigue graph is greatest when the temperature gradients are steepest.

Even for water-cooled strain-hardened material of moderate thermal conductivity, such as the strain hardened nickel represented in Fig. 1, the distorting influence of the fatigue graph is apparent. This suggests that even for such material thermal influences due to hysteresis may play a much larger part than is usually supposed in giving form and position to the ordinary fatigue graph. It seems not improbable, therefore, that the actual temperatures in a metal under fatigue are considerably higher than has been supposed.

The distortion of the corrosion-fatigue graph above the endurance limit, therefore, may be due to elevation of the effective temperature. For alloys, such as those represented in Fig. 5, in which the steep portion of the corrosion-fatigue graph is well to the left of the corresponding portion of the fatigue graph, the number of cycles to failure under corrosion-fatigue is so few that elevation of temperature in the specimen would have relatively little effect on the form of the graph. Even under these conditions, however, as shown in Fig. 5, there are in the upper portions of the corrosion-fatigue graphs some indications of straightening and shifting toward the left, though there is no clear evidence of reversal of curvature as in the complex graphs of Figs. 1 to 3.

This hypothesis that distortion of the corrosion-fatigue graph is due largely to the thermal effects of ordinary fatigue, has not yet been definitely established. More light may be thrown on the subject by experiments now under way at lower cycle frequency.

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[For Discussion on Fatigue of Metals, see page 134.—ED.]

EFFECT OF CORROSION UPON THE FATIGUE RESISTANCE OF THIN DURALUMIN

By R. R. Moore[1]

Synopsis

This paper gives the results of fatigue tests on thin gage corroded duralumin. The investigation was made to determine the effect of the embrittlement of duralumin, due to corrosion, in terms of the resistance of the metal to repeated stresses or fatigue.

The results show the effect of a certain degree of corrosion upon the endurance limit. The corrosion of duralumin occurs predominately along the grain boundaries ("intercrystallin") and is of a continuous or progressive nature rather than a local pitting type, although some of the latter is present.

A method of making accurate fatigue tests on thin-gage light metals is given and the machine used in these tests is described.

It has been known for a long time that duralumin is subject to corrosion. Thin-gage duralumin sheet in particular is rendered very brittle by such corrosive action. The significance of this embrittlement in terms of its effect upon the metal's resistance to repeated stresses or vibration has not, to the author's knowledge, been determined. The present tests were made to determine the effect of corrosion upon the endurance limit of thin duralumin.

Method of Test

Testing Machines and Specimens:

Apparently, there is no machine commercially available which is sufficiently reliable for accurate fatigue tests on thin sheet stock of the light alloys. The author has overcome this difficulty by using thin wall tubes instead of sheet, the tests being made in the rotating beam type of fatigue machine. It was necessary, however, to use a special type of rotating beam machine developed by the author and shown in Fig. 1. This is a "short specimen" type of machine which is essential for testing tubes. The principal features of the machine are that it is very accurate, smooth running, easy to set up and employs a short inexpensive specimen. It consists essentially of two aluminum housings which pivot on fulcrum pins as shown. Each housing is bored and babbitted to support a hardened steel spindle. The end of each spindle is taper-bored to receive the tapered end of the fatigue specimen. The specimen ends are drawn snugly into these tapered holes by means of a specially designed bolt arrangement. The load is applied through hangar yokes which are supported on knife edges set at a fixed distance from the fulcrum pin. The stress in the specimen is calculated by means of the beam formulas.

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[1] **Chief, Physical Testing Branch, U. S. Air Corps, McCook Field, Dayton, Ohio.**

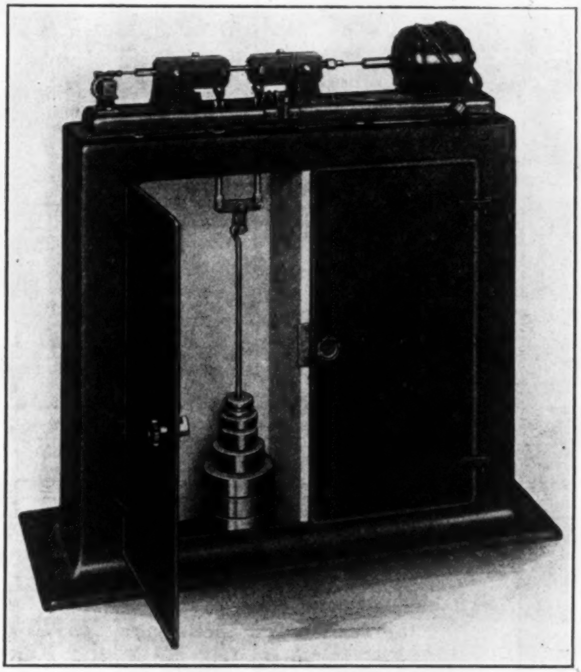
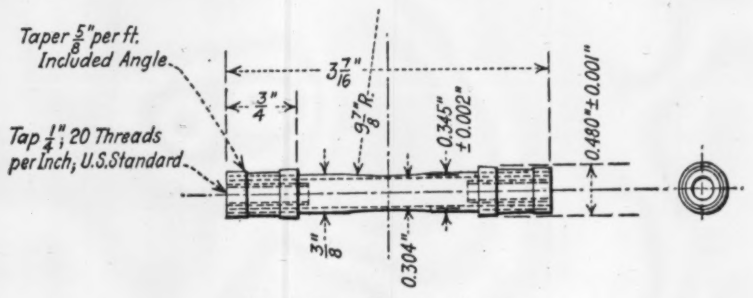


FIG. 1.—Fatigue Testing Machine.

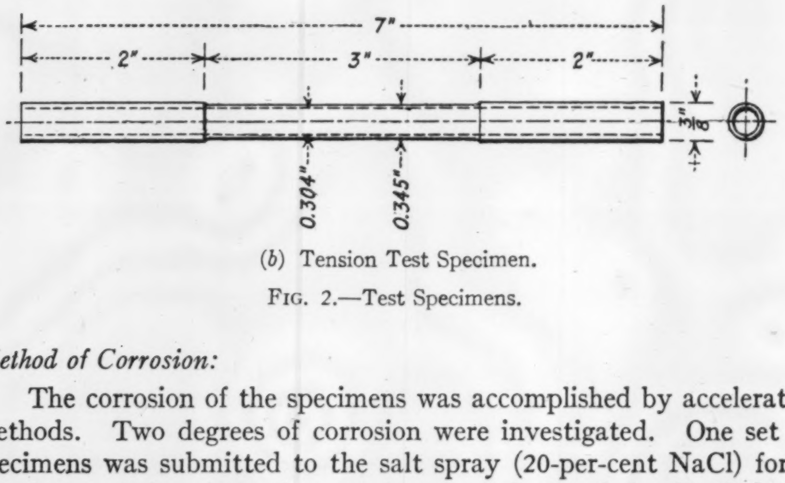
The fatigue-test and tension-test specimens are shown in detail in Fig. 2. These specimens were made from duralumin (17ST) tubing $\frac{3}{8}$ in. in diameter and 0.035 in. in wall thickness. The chemical analysis of the duralumin is as follows:

Copper	4.26	per cent
Magnesium	0.55	"
Iron	0.23	"
Silicon	0.21	"
Manganese	0.49	"
Aluminum (by difference)	94.26	"

The tension tests were made in a standard Olsen 20,000-lb. testing machine arranged for 2,000-lb. maximum capacity. Special self-aligning grips were used to test these specimens. These grips were developed by R. L. Templin of the Aluminum Co. of America and are shown in Fig. 3. In making tension tests on light-alloy thin-wall tubes it is absolutely necessary to use a light self-aligning grip in order to obtain accurate elongation values. If the specimen is not axially loaded it will tear prematurely, thus affecting the elongation.


Taper 5/8" per ft. Included Angle
Tap 1/4", 20 Threads per Inch; U.S. Standard
3 7/16"
3/4"
9 7/8" R.
0.345" ± 0.002"
0.480" ± 0.001"
3/8"
0.304"

(*a*) Fatigue Test Specimen.



(*b*) Tension Test Specimen.

FIG. 2.—Test Specimens.

Method of Corrosion:

The corrosion of the specimens was accomplished by accelerated methods. Two degrees of corrosion were investigated. One set of specimens was submitted to the salt spray (20-per-cent NaCl) for a five-day period and another set for a ten-day period. During the five-day period the spray only operated eight hours per day and was off for 16 hours. During the ten-day period the spray was operated continuously night and day for the first two days; then intermittently for the next six days, that is, on and off every hour for eight hours and then off sixteen hours; for the next two days the spray operated continuously.

RESULTS OF TESTS

The endurance curves are given in Fig. 4. The endurance limits obtained from these curves together with the results of the tension tests are given in Table I. All values excepting the endurance limits are the average of three tests.

It is evident that the proportional limit and tensile strength are unaffected by the corrosion.

The ductility, however, is affected considerably. There is a loss of 25 per cent in ductility due to corrosion. Both the five and ten-day periods show practically the same effect. It can be said that the ten-day specimens were more consistent, as each of the three specimens tested showed 12.5 per cent elongation, whereas the five-day specimens gave values of 11.3, 13.3 and 12.5 per cent. These results, however, are so close that their differences have no significance.

The results of the fatigue tests are interesting, for so far as the author is aware, they are the first published tests to show the effect of corrosion on the fatigue of duralumin. In general, the effect of the degrees of corrosion experimented with was to decrease the endurance limit about 35 per cent. The difference between the effects of the five and ten-day corrosion is very small.

The effect of the corrosion experimented with was to reduce the endurance limit to a minimum of 11,000 lb. per sq. in. It is not to be concluded that this is the minimum ever to be expected in service. It is highly probable that if the corrosion is allowed to progress as the metal is being repeatedly stressed (a practical service condition), the effect of this "progressive corrosion" will be more severe. It should be particularly noted that the tests given in this paper are what may be called "preliminary corrosion" fatigue tests.

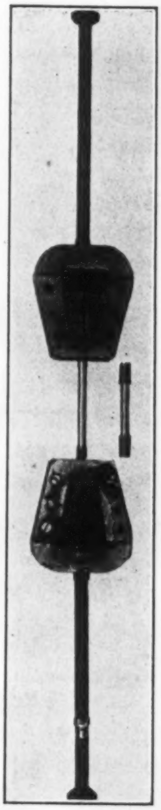


FIG. 3.—Self-Aligning Grips for Tension Test.

Attention is directed to the fact that the static tensile strength is unaffected by the corrosion, whereas the resistance to repeated stresses is considerably affected. The repeated-stress or fatigue test is evidently considerably more sensitive to certain factors than is the ordinary tension test, and for that reason it is a more valuable test in determining the serviceability of the metal.

METALLOGRAPHY

In Fig. 5(*a*) is shown a photomicrograph that is representative of the structure of the duralumin tubes.

A micrographic survey of the specimens corroded by both the five and ten-day periods showed no marked difference in the corrosion effects of either method. Evidently, the stopping of the spray over night in the five-day method gave considerable opportunity for the corrosion to progress, and resulted in a degree of corrosion practically as severe as the six days of intermittent corrosion experienced in the ten-day method.

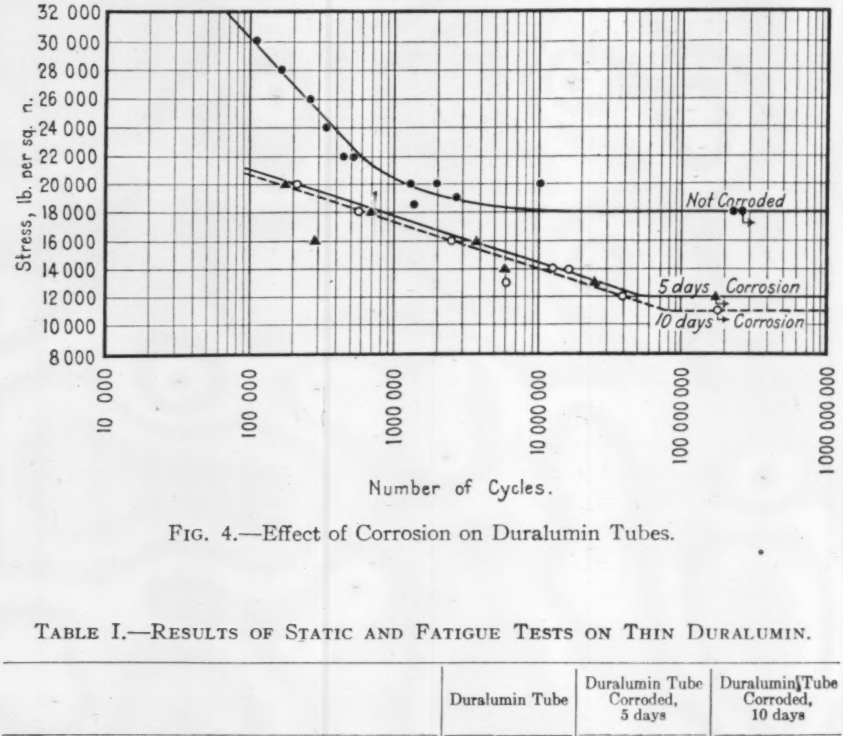
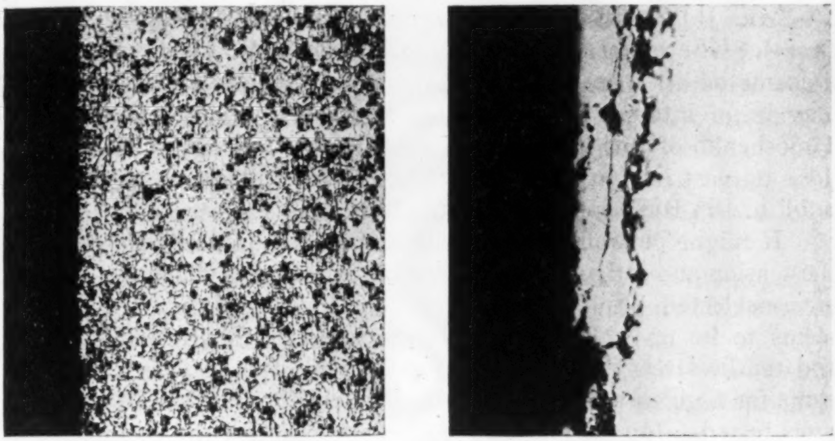


FIG. 4.—Effect of Corrosion on Duralumin Tubes.

TABLE I.—RESULTS OF STATIC AND FATIGUE TESTS ON THIN DURALUMIN.

	Duralumin Tube	Duralumin Tube Corroded, 5 days	Duralumin Tube Corroded, 10 days
Outside Diameter, in	0.344	0.344	0.344
Inside Diameter, in	0.304	0.304	0.304
Proportional Limit, lb. per sq. in	40 635	39 410	39 410
Tensile Strength, lb. per sq. in.	69 400	67 050	67 930
Elongation in 2 in., per cent	16.8	12.4	12.5
Endurance Limit, lb. per sq. in	18 000	12 000	11 000
Ratio, Endurance Limit to Tensile Strength	0.26	0.18	0.16

In Fig. 5(*b*) is shown a sample of the ten-day corroded material unetched. Note particularly that the corrosion is not of the local pitting type but is predominately of a progressive nature. Detail study shows that it generally follows the grain boundaries. For this reason it has been termed "intercrystallin" corrosion and is distinguished from the pitting type of corrosion usually associated with the rusting of steel. This photomicrograph shows the edge of the thin duralumin tube wall so that, bearing in mind the 500 magnification, an idea is obtained as to the relative penetration of the corrosion. The long fine crack which is intersected by a large sub-surface cavity lies about 0.0004 in. below the outer surface of the tube wall. This is about 2 per cent of the total thickness of the metal.



(*a*) Representative Structure, Longitudinal. Etched for 5 seconds in Flick's Reagent (HCl 13 per cent; Concentrated HF 8.7 per cent; Water 78.3 per cent). (× 100.)

(*b*) Material Corroded for 10 Days, Longitudinal. Unetched. (× 500.)

FIG. 5.—Photomicrographs of Structure of Duralumin Tubes.

DISCUSSION ON FATIGUE OF METALS

FATIGUE OF CAST IRON

Mr. Lessells. MR. J. M. LESSELLS[1] (*presented in written form by Mr. P. L. Irwin*).—The paper by H. F. Moore and S. W. Lyon will form a welcome addition to the literature on the strength of cast iron. It may be of interest to the authors to know that according to Mr. Timoshenko of this laboratory a considerable amount of testing was done on cast iron at Kieff Polytechnical Institute by Voropaeff and published in Bulletins of the Institute in 1914.

It might be well to point out certain features in the results of the tension and fatigue tests at normal temperature which the authors have neglected. In the first place (see Table II of the paper), there seems to be no well defined relation between the Brinell hardness and tensile strength for cast iron since while this ratio is fairly constant for a given analysis it changes widely for the various compositions tested. Furthermore the endurance ratio for the Grade No. 91 which was centrifugally cast is much higher than that for the other materials. This may be an advantage obtained by the centrifugal type of casting. It is strange, as may be concluded from remarks on page 88, that the micrographs showed no difference in this casting. It is also stated that the tension tests showed no clearly marked elastic limit. The writer wonders if this may be due to the particular grade of cast iron or rather to the method of measuring extensions. From tests made in the Research Laboratory of the Westinghouse Co., we have shown that cast iron has an elastic limit in compression. It is however very low and very sensitive extensometers must be employed. The tensile strength and endurance limit for the same material were 27,800 lb. per sq. in. and 13,800 lb. per sq. in., respectively.

The results of understressing are certainly very interesting. The increase in fatigue strength encountered in rolled steel by this understressing may be due to a progressive strain hardening. Do the authors feel that the same has taken place with the cast iron?

The effect of the groove is also of interest. The writer feels however that cast iron is not the kind of material to examine if we want to throw further light on the effect of ductility on stress concen-

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[1] Engineer in Charge, Mechanics Section, Research Laboratory, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

Mr. Lessells. tration. The problem is complicated enough using the simpler materials and much testing has yet to be done before a true explanation will be forthcoming.

The tests at high temperature call for no special comment. It is a pity however that delicate extensometers were not used in the tension tests so that some data on the modulus of elasticity could have been obtained. The fact that cast iron retains its strength with increase in temperature as brought out in the paper is well known.

The results of cycles of stress from zero to a maximum are interesting. They seem to indicate that the fatigue range for a material whose compressive strength exceeds its tensile strength continues to increase as the mean stress assumes higher negative values. This seems to support the results Haigh obtained on brass,[1] but further tests on this phase of the subject will be necessary before final conclusions can be drawn.

Mr. Kommers. MR. J. B. KOMMERS[2] (*presented in written form*).—The paper by Moore and Lyon is a very interesting contribution in a field in which very little information on fatigue strength is available.

The most striking part of the paper to me was the series of tests on range of stress. When the stress in the Illinois tests was entirely compressive from zero to a maximum, the range of stress was 65,000 lb. per sq. in. When a small amount of tension was introduced into the cycle so that the range of stress was from 32,000 lb. per sq. in. compression to 6400 lb. per sq. in. tension the range was immediately reduced from 65,000 to 38,400 lb. per sq. in. When more tensile stress was introduced into the cycle so that the range was from 10,500 lb. per sq. in. in compression to 10,500 in tension, the range was still further reduced to 21,000 lb. per sq. in. When the stress was entirely tension with the minimum stress zero and the maximum stress 15,500 lb. per sq. in. in tension, the range was the smallest of all, being only 15,500 lb. per sq. in.

The range of stress, therefore, in these tests varied from 65,000 lb. per sq. in. when the stress was entirely compression, to 15,500 lb. per sq. in. when the stress was entirely tension. It is evident, therefore, that for cast iron tensile stress is especially effective in producing fatigue.

Another interesting matter mentioned in this paper is the fact that cast iron may have its endurance limit increased by understressing. This result need not be surprising because it has been shown that a material as brittle as concrete may have its endurance limit

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[1] B. P. Haigh, "The Fatigue of Brass," *Journal*, Institute of Metals, September, 1917.

[2] Associate Professor of Mechanics, University of Wisconsin, Engineering College, Madison, Wis.

Mr. Kommers.

increased in the same way. I speak of concrete in this connection because it, like cast iron, also has a low tensile strength and a high compressive strength. Furthermore, even in an ordinary fatigue test of concrete in compression, the first application of stress produces a curved stress-deformation curve, which becomes a straight line under continued stressing. In this process of adjustment, the modulus of elasticity of the concrete is decreased. It would be interesting to determine whether cast iron also has its modulus of elasticity decreased while it is adjusting itself to a cycle of stress under which ultimate failure does not occur.

One of the matters which should be seriously considered by investigators of fatigue phenomena is the matter of range of stress, for steel, for cast iron, for non-ferrous metals, and for concrete. Our information is not at all complete, and should be made so as soon as possible. Mr. McAdam has done some work on range of stress in torsion, and Mr. H. F. Moore and his associates have done work on cast iron and on certain steels in tension-compression. Mr. Haigh, in England, did some work on range of stress for brass in tension-compression.

These tests on range of stress are of course much more difficult to carry out than ordinary rotating-beam tests. Probably direct stress experiments in tension-compression would be the most satisfactory. But these tests should not be attempted unless a machine is used in which there will be no eccentric loading in the compression portion of the cycle. Mr. P. L. Irwin of the Westinghouse Co. in a paper before this Society two years ago showed us how this could be accomplished. This work was important not only because of the device which he developed for producing truly axial stress, but also for the fact that he proved that rotating-bending and axial stress give the same endurance limit. I hope that tests on range of stress will be carried out in the near future, in spite of the fact that it will be a time-consuming task.

Mr. R. R. Moore.

MR. R. R. MOORE[1] (*by letter*).—The writer is particularly interested in what Moore and Lyon have called in their paper, "strain adjustability." It appears that strain adjustability may be studied from two viewpoints, namely, the effects of notches (stress raisers) and by understressing or coaxing tests. In regard to the effects of notches, the writer presented a paper before this Society in 1925[2] in which it was shown that the notch effect was independent of elongation. These tests covered a variety of materials such as carbon steel, magnesium-aluminum alloys, magnesium-copper alloys, pure mag-

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[1] Chief of Physical Testing Branch, U. S. Air Corps, McCook Field, Dayton, Ohio.

[2] R. R. Moore, "Some Fatigue Tests on Non-Ferrous Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 66 (1925).

Mr. R. R. Moore. nesium, naval brass, and aluminum bronze cast and extruded. These tests showed also that cast aluminum bronze developed less notch effect (and probably more strain adjustability) than the other alloys. It is interesting to compare this with the similar results of Moore and Lyon on cast iron.

CORROSION-FATIGUE

Mr. McAdam. MR. D. J. MCADAM, JR.[1]—In discussing the paper by R. R. Moore, I wish to present two lantern slides[2] which give a comparison of corrosion-fatigue and prior-corrosion fatigue. By corrosion-fatigue I mean simultaneous corrosion and fatigue. By prior-corrosion fatigue I mean stressless corrosion in water followed by fatigue in air.

Comparison of the effects of corrosion-fatigue and prior-corrosion fatigue on duralumin has been made at the Naval Engineering Experiment Station. The results show that prior-corrosion fatigue is much more damaging for duralumin, and for other aluminum alloys to be described elsewhere, than for steel or non-ferrous alloys having lower electrolytic solution pressure. The results of prior-corrosion fatigue, obtained with the tapered specimen that has been previously described, agree fairly well with the results obtained by Mr. Moore.

It seems possible that the high solution pressure of aluminum alloys is the general cause of the susceptibility of duralumin and other aluminum alloys to prior-corrosion fatigue.

I note that Mr. Moore now obtains a curved graph for duralumin. This differs in form from the graphs presented by Mr. Moore in his first paper on duralumin. At the Naval Experiment Station, we have usually obtained the curved type of graph, but one sample of duralumin recently tested gave a straight sloping graph extending to about 100,000,000 cycles before beginning to approach a horizontal asymptote. I have come to the conclusion that samples giving this abnormal type of graph are for some obscure reason unusually subject to corrosion-fatigue in air. It is possible that all duralumin is subject to some extent to corrosion-fatigue in air.

Mr. R. R. Moore. MR. R. R. MOORE (*author's closure by letter*).—The lantern slides referred to by Mr. McAdam were not available to the author and therefore could not be examined and commented upon. Mr. McAdam makes a reference to the author's previous graphs for duralumin, but

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[1] Metallurgist, U. S. Naval Engineering Experiment Station, Annapolis, Md.

[2] The two lantern slides referred to in this discussion showed a very complicated system of graphs. The publication of these graphs would not be desirable unless they were accompanied by a long, detailed discussion or explanation. They have accordingly been omitted. They appear together with the discussion thereon in a paper by D. J. McAdam entitled "Fatigue and Corrosion-Fatigue of Metals," presented at the Amsterdam International Congress on Testing Materials.—ED.

Mr. R. R. Moore.

this matter has been thoroughly discussed elsewhere,[1] and needs no further comment. Furthermore, Mr. McAdam admits in his last sentence that all duralumin may be subject to some corrosion-fatigue which will affect the shape of the graph. This thought has been presented by the author in a discussion of Mr. McAdam's paper "Corrosion-Fatigue of Non-Ferrous Metals," and will be found elsewhere in these Proceedings.[2]

Mr. Lessells.

MR. LESSELLS (*presented in written form by Mr. P. L. Irwin*).—The paper by Mr. McAdam forms a further interesting contribution on the influence of corrosion on fatigue of metals. It is to be regretted, however, that the paper, while it does provide data on the strength of these non-ferrous metals for the prescribed conditions which are of course valuable to the designer, it does not give us sufficient information on the mechanism of failure. There are so many variables and the paper and those which preceded it cover such a range of materials that it is to be hoped the author will now give us a more complete analysis of his results.

In the first place, it is probably misleading to say (page 121) that the corrosion-fatigue limit for steels varies surprisingly little with composition and heat-treatment. While this is substantially true, it might be better to talk about the reduction in fatigue strength due to corrosion since this will give an entirely different picture. Referring to Table V of a paper by McAdam entitled "Corrosion-Fatigue of Metals,"[3] presented before the American Society for Steel Treating, it will be found that the endurance limit for 0.11-per-cent carbon steel is lowered by corrosion from 25,000 lb. per sq. in. to 16,000 lb. per sq. in. and for a 0.36-per-cent carbon steel this lowering is from 52,000 lb. per sq. in. to 19,000 lb. per sq. in.

As regards the effect of cold working on steels we do know[4] that the tendency to corrode is increased by the degree of cold working. Considering then the results obtained for pure nickel, we see that while the corrosion endurance limits for the cold-worked and annealed materials are alike, the percentage reduction for the cold-worked material is considerably greater. This may be explained in a similar manner to steels.

A further point which may be of interest is the apparent effect of nickel on corrosion. Referring to the previous paper, it was found that the endurance limit for a 5-per-cent nickel steel was reduced

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[1] D. J. McAdam, Jr., "Endurance Properties of Non-Ferrous Metals, Part II," Paper 1537D, Am. Inst. Mining and Metallurgical Engrs., issued with *Mining and Metallurgy*, February, 1926.

[2] See p. 151.

[3] D. J. McAdam, Jr., "Corrosion-Fatigue of Metals as Affected by Chemical Composition, Heat Treatment, and Cold Working, Part I," *Transactions*, Am. Soc. Steel Treating, March, 1927, Vol. XI, No. 3.

[4] Goerens, Carnegie Scholarship Memoirs (Iron and Steel Inst.), 1911, Vol. 3, p. 320.

Mr. Lessells. from 66,000 lb. per sq. in. to 19,000 lb. per sq. in. by corrosive effects. In the present paper, for the nickel-copper alloys it may also be noted that for the EI series as annealed where the nickel is approximately 48 per cent, the values are 32,000 lb. per sq. in. and 24,000 lb. per sq. in., whereas for the HE series as annealed where the nickel is approximately 21 per cent they are both 18,000 lb. per sq. in.

These observations may serve to illustrate what the writer has in mind, namely, that in this problem there are evidently many variables, either those of a mechanical nature such as cold working or those of a chemical or metallurgical nature due to material composition or to corrosive media. In this latter respect, while no effects were found for the copper with the agents used, this does not exclude, of course, the possible effects of other agents. Probably this question of corrosion is one which can best be approached by the study of single crystals similar to what is now being done in fatigue.

As regards the possible influence of the elastic limit in tension on the corrosion-fatigue limit, as suggested on page 122, there may be something to this. It should, however, be noted that cases have previously been given (see previous reference), for example, chrome-nickel steel FL where the endurance limit was 50,000 lb. per sq. in. and the proportional limit 35,200 lb. per sq. in., but the corrosion-fatigue limit was also 50,000 lb. per sq. in. So while it seems feasible that the deformation may influence the action of corrosion, this does not appear to be supported by the results obtained, since in the above case the endurance limit was considerably above the proportional limit and excessive deflection could have been expected; nevertheless the corrosion-fatigue limit was unaffected.

Mr. Templin. MR. R. L. TEMPLIN[1] (*presented in written form*).—Mr. McAdam is to be complimented on this recent addition to his very extensive studies of fatigue phenomena and properties of metals. Referring to the results which he obtained from tests of duralumin, however, it should be pointed out that the producers of the material do not consider the metal which he has designated as "GT" simply another temper or condition of the metal designated as "DU." The metal "GT" is known by the trade designation Special 17*S*-*T* and the other as simply 17*S*-*O*. The apparently slight differences in chemical composition of the two alloys are sufficient to account for the rather high tensile strength found for the Special 17*S*-*T* as compared to normal 17*S*-*T*. To the producers of these alloys, therefore, it does not seem quite proper to compare their fatigue properties as though they were simply different tempers of the same alloy.

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[1] Chief Engineer of Tests, Aluminum Co. of America, New Kensington, Pa.

TABLE I.—COMPARISON OF FATIGUE DATA ON ALUMINUM AND ALUMINUM ALLOYS.

	Stress at 1 000 000 Cycles	Stress at 10 000 000 Cycles	Stress at 100 000 000 Cycles	Endurance Limit, lb. per sq. in.	Approximate Number of Cycles Determining End Limit
MATERIAL 2*SH*					
McAdam............	11700	9500	8500	8400	170 000 000[a]
Aluminum Co. of Am.	11800	9500	8500	8000–8500	500 000 000[b] stressed at 8000 lb. per sq. in.
MATERIAL $2S\frac{1}{2}H$					
McAdam............	9600	8000	7300	7000	100 000 000[a]
Aluminum Co. of Am.	10000	8500	7600	7000–7500	350 000 000[b] stressed at 7000 lb. per sq. in.
MATERIAL 3*SH*					
McAdam............	14500	11700	11000	10800	100 000 000[a]
Aluminum Co. of Am.	14000	11500	10500	10000–10500	450 000 000[b] stressed at 10000 lb. per sq. in.
MATERIAL $3S\frac{1}{2}H$					
McAdam............	13000	10500	10100	10000	100 000 000[a, c]
Aluminum Co. of Am.	12500	10500	9800	9000–9500	400 000 000[b] stressed at 9000 lb. per sq. in.
MATERIAL 3*SO*					
McAdam............	7700	7100	7500	7000	100 000 000[a, c]
Aluminum Co. of Am.				7000–7500	450 000 000[b] stressed at 7000 lb. per sq. in.
MATERIAL 17*S–T*					
McAdam[d]...........	26000–30000	19000–22000	16000–18000	16000–18000	100 000 000[a]
McAdam............	26000–28000	19500–22000	16500–18500	16000–18000	200 000 000[b] stressed at 16000 lb. per sq. in.
Aluminum Co. of Am.	28000	21700	17000	15000–15500	250 000 000[a] stressed at 16000 lb. per sq. in.
MATERIAL 25*S–T*					
McAdam............	25000–27000	18000–20000	15500–17500	15000–17000	140 000 000[a] stressed at 15000 lb. per sq. in.
Aluminum Co. of Am.	26000	21000	16500	15000–15500	350 000 000[b] stressed at 15000 lb. per sq. in.
MATERIAL 51*S–T*					
McAdam............	18000	13200	12500	12500	50 000 000[a]
Aluminum Co. of Am.	21000	16000	12000	10500–11000	330 000 000[a]

[a] Specimen not broken. [b] Specimen broken. [c] Accelerated test (stress raised at intervals).
[d] Special 17*S–T*.

NOTE: **McAdam's tests made using rotating cantilever machine.**
Aluminum Co. of America's tests made using R. R. Moore's rotating beam testing machine.

Mr. Templin. Similar fatigue but not corrosion-fatigue tests have been made in the General Laboratory of the Aluminum Co. of America, on duplicate samples of some of the alloys tested by Mr. McAdam and a comparison of the results obtained in the two laboratories may be of interest, bearing in mind that Mr. McAdam uses a specimen and testing machine as he has indicated, whereas we have used R. R. Moore machines and specimens. Some of the test results which may be compared are given in Table I.

Mr. McAdam has given us some interesting suggestions as to the causes of early fatigue failure under corrosive conditions. It is rather disconcerting to be confronted with facts apparently showing that the much enhanced static tensile properties of heat-treated metals may be of doubtful advantage if the parts made from such materials are subjected to corrosion-fatigue conditions of stress. This but serves to emphasize the question, what is the primary factor or factors that determine the fatigue strength of a metal.

Mr. Gillett. MR. H. W. GILLETT[1] (*presented in written form by the Assistant Secretary*).—This continuation of Mr. McAdam's work on corrosion-fatigue gives results in line with his previous ones and again leads him to the major conclusion that corrosion-fatigue depends almost wholly on chemical composition. If this conclusion be accepted, then heat treatment or cold working for increasing the strength of metals and alloys is labor lost if corrosion-fatigue conditions are those which govern in service.

Findings so revolutionary reflect great credit on Mr. McAdam for his discovery and put a responsibility on designing engineers to utilize the facts brought out by Mr. McAdam just as soon as those facts are sufficiently substantiated for general acceptance.

A responsibility is also placed on other workers in the fatigue field to repeat and amplify Mr. McAdam's tests so that substantiation will be prompt. This may sound like advocating wasteful duplication of effort, but it is in reality the opposite of waste. McAdam's conclusions on corrosion-fatigue affect and change the very foundation of engineering design of all structures in which corrosion-fatigue can play a part. When building foundations, it is best to make them broad and solid.

In ordinary fatigue work, the facts and conclusions upon which workers like McAdam, H. F. Moore, R. R. Moore, Gough and Lea, for example, all agree, have to-day a value for engineering design that they would not have had before ample corroboration had been obtained. Similarly, the value of McAdam's work on corrosion-fa-

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[1] Chief, Division of Metallurgy, U. S. Bureau of Standards, Washington, D. C.

Mr. Gillett. tigue will be multiplied when it is repeated by others with different sizes of specimens and different types of testing machines.

Lehmann[1] has just reported a series of fatigue tests on 0.33-per-cent carbon steels as follows:

TEST CONDITIONS	ENDURANCE LIMIT, LB. PER SQ. IN.
In air at 17° C.	±41 000
In distilled water at 96° C.	±38 500
In sodium chloride solution at 96° C.	±41 250
In sodium nitrate solution at 96° C.	±39 000
In ammonium chloride solution at 96° C.	±32 500

McAdam's tests[2] on 0.24 and 0.36-per-cent carbon steels gave in air at room temperature ±27,000 and ±34,000 lb. per sq. in. endurance limits, respectively, and fell to ±16,000 and ±25,000 lb. per sq. in. endurance limits, respectively, in fresh water at room temperature. It is hard to reconcile the results of the two workers. Certainly one would not predict from McAdam's tests that it would be possible for Lehmann to test a steel in hot salt solution and find a higher endurance limit than in hot distilled water or even than in air at room temperature. What is the poor designing engineer to do when confronted by such conflicting information, save ask for enough more data to explain the discrepancies?

On account of the very tentative and conservative way in which McAdam has phrased his theoretical discussion, it is assumed that some, at least, of the double-branched curves in the plots are also to be taken as tentative.

Certainly in Fig. 3 of the paper, the dashed curves of HE-4, EI-14 and EI must have been drawn on the basis of McAdam's expectations of their course, based on analogy, since the scatter of the points hardly justifies the branched curves. In the first and last, a straight line represents the open triangles as well as the curves, and in all three plots the dashed curves seem to be extrapolated beyond experimental justification by the points plotted. These particular plots may very well represent the true facts, but it would be interesting to see how those curves would be drawn by an engineer who had only the plotted points and no further information on the expected course of the graphs.

On page 123 reference is made to the "amorphous metal" of grain boundaries and slip lines. The argument is the same if reference to amorphous metal is omitted, and since the existence of metal that can be termed "amorphous" in any strict sense of that word is becoming more and more doubtful, it would be well, if that explanation is to be retained, to define the author's conception of "amorphous" metal. **Mr. Gillett.**

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[1] G. D. Lehmann, "The Variation in the Fatigue Strength of Metals when Tested in the Presence of Different Liquids," British Aeronautical Research Committee, Reports and Memoranda No. 1054, October, 1926.

[2] D. J. McAdam, Jr., "Corrosion-Fatigue of Metals, Part II," *Transactions*, Am. Soc. Steel Treating, 1927.

But it is less important at this stage to develop a theory to account for the observed phenomena than it is to corroborate the observations of the phenomena. Engineers certainly hope that McAdam's conclusions are not true, and equally certainly, they are afraid that they are true! The bad news that the resistance of our metallic materials of construction is so low under conditions of combined repeated stress and corrosion ought to be verified beyond all possibility of argument just as rapidly as it can be done.

The more one thinks about the problem of corrosion-fatigue the more he is impressed by the service Mr. McAdam has done in focusing attention upon it.

Mr. McKay. MR. R. J. MCKAY.[1]—Mr. McAdam has opened a new door of knowledge to the student of the properties of materials. A relation between the corrosion of metals and their action under stress, is thermo-dynamically necessary. Mr. McAdam has demonstrated this relation experimentally, where the experiments of many other workers have been inconclusive. I hope the present observations from the standpoint of a student of corrosion may aid in making the great practical value of these results rapidly available to engineering design.

Failure of the specimen in McAdam's test is caused by the combined effect of a repeated stress of definite proportions with a corrosive action which is gradual. McAdam apparently believes that the failure depends largely on the rate of corrosion. If this is true, it must also depend upon the time of application of this corrosion rate. Therefore, we might expect that the corrosion-fatigue limit would be different for different rates of testing, and if so, the corrosion fatigue limit in practice would depend on the time during which the metal is stressed. Possibly the convex and concave portions of McAdam's curves are due to these competing rates of corrosion and fatigue; and therefore some of the corrosion-fatigue curves might be even more complicated than those he has drawn.

It is valuable to visualize a definite corrosion-fatigue limit which may be stated in terms of pounds per square inch. However, it might be also useful to consider this corrosion-fatigue limit rather as a function of the corrosion resistance of the metal and of its strength properties. This seems a practical method of procedure, because the corrosion resistance of the metal is a function of several variations, and

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[1] Superintendent, Technical Service, International Nickel Co., New York City.

Mr. McKay. to define these variables to make them constant, and thus obtain a single quantitative value, means that the value obtained will not represent any wide field of application to design. For instance, considering the solution which Mr. McAdam has used, these have been admirably selected. It would obviously be necessary, however, to define the oxygen contents and the rate of motion past the surface of the sample, to define the corrosion resistance of the material in these solutions, and there are, in addition, many other solutions to consider.

In regard to Mr. McAdam's suggestion of a possible relation between electrolytic potential of nickel-copper alloys and their corrosion-fatigue limits, this is presented in a very open-minded way, and it is obviously still anybody's guess as to whether or not the two matters are connected. The results of Gordon and Smith, on which McAdam bases this interesting relation, should, I believe, be interpreted differently. It is true that the variation figure shown, with the difference 0.585 minus 0.563 volts equalling 0.022 volts, looks small. I should like to point out that in other parts of Gordon and Smith's experiments, they show a variation of about twice that, and that in all cases this variation is distinct and reproducible, and is a function of the nickel-copper ratio of the alloys. In other words. it is a true variation, and cannot be considered as an equality, though the differences may look small. The reason they look small is because we have been taught to consider the variation of potential of alloys as taken in solutions of their own salts, and we too often lose sight of the effect that would be obtained were we to use a series of metals or alloys all in the same solution. The potential of an electrode depends not only on the electrode, but very much more on the solution with which it is in contact. The variation shown here for nickel-copper alloys, is not unusually small for alloys in solutions of the same metal ions. In this case the copper ion and its concentration is the controlling agent of the single potential of the electrodes. Gordon and Smith show the potential to vary uniformly with the copper concentration in the solution, and also uniformly with the copper-nickel ratio of the alloy, and give conclusive proof that both of these variations concur closely with the theoretical values as calculated from Nernst's thermodynamic relation. Thus I believe that the electrode potential of these alloys varies, as is to be expected, and is not equal in the same sense as the practical similarity of the corrosion-fatigue limits.

A more promising theory seems to be that of some material present in all these alloys which is attacked at a rate very much the same in all these tests. Mr. N. B. Pilling has pointed out to the speaker that the same constituent may be expected to be present in all these alloys and that it might be a point of attack, and Mr. Merica has pointed out that the equilibria of the Gordon and Smith experiments was probably with a modified alloy surface and not with the original alloy, also that such effects as loss of ductility during corrosion are very specific both with regard to alloys and to solutions. **Mr. McKay.**

From these considerations, I would draw the conclusion that, in addition to the program of tests suggested by Mr. McAdam, namely an investigation of additional series of binary alloys, tests should be made on the metals on which he already has data, using a more varied range of corrosive agents. This would bring the tests made up to the present immediately into the field of practical use and add as much to scientific knowledge. Tests including wide variations in corrosion rates, and also in types of corrosion, would seem practical, including corrosion of actual zero amount.

Mr. Speller. Mr. F. N. Speller.[1]—As time goes on, corrosion-fatigue certainly becomes more interesting. It is getting close to some practical everyday problems. The effect of variable external conditions should perhaps be the next step in this investigation, and I have no doubt Mr. McAdam has that in mind. He says something about having made tests with the application of protective coatings, but the coatings do not seem to me to be entirely protective. They would be considered rather imperfect by some. Would it not be possible to use something like a rubber coating that would absolutely exclude air, and thus get zero corrosion for one series of tests? We are planning to make some tests with water carrying various inhibitors.

The corrosion-fatigue tests, generally speaking, seem to be in line with the results of immersion tests on ferrous alloys with respect to the effect of heat treatment and composition, in that for the same class of steel these variables do not seem to be important factors. That may be of some significance.

The short time required to cause failure is a difficult thing to explain considering the little corrosion that is visible on the test piece. However there may be some local increase in temperature at the point of maximum stress, together with the so-called concentration cell effect in surface crannies, which increases the rate of corrosion very rapidly, and more particularly the maximum rate of corrosion may be maintained at the base of these crannies or cracks at all times due to alternated stresses preventing the formation of natural protective coatings. | These three factors all tend to maintain a maximum

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[1] Metallurgical Engineer, National Tube Co., Pittsburgh, Pa.

Mr. Speller. rate of corrosion, and therefore might explain the marked effect of corrosion under such conditions of stress in very short periods of time. We have shown before that when iron is first immersed in water the metal tends to flash into solution at a maximum rate, and that, in a space of five or ten minutes, the rate of corrosion is often reduced to less than 20 per cent of the original rate;[1] if it happens to be an inhibiting solution, the rate of corrosion is quickly reduced to a much smaller figure. It is easy to imagine, therefore, when the stresses are rapidly alternated, that there is no chance of a protective film forming, and therefore the maximum rate of attack would be maintained. I should like to ask Mr. McAdam for a little more information about the composition of the water used in these tests. He told us this morning that the salt water was the brackish water of the Severn River; that is the water that Committee A-5 on Corrosion of Iron and Steel uses in making immersion tests at Annapolis and which is not particularly corrosive—not so corrosive as Washington city water, for instance. As for the fresh water, with 11.6 grains per gallon of calcium bicarbonate, this water would certainly not be considered a very corrosive water. I cannot help feeling that the rapid effect of what is called corrosion-fatigue is due to the prevention of the formation of the usual surface protective films.

Mr. Smith. MR. H. E. SMITH.[2]—I wish to mention just one connection these laboratory tests may have with the operation of steam boilers. For many years it has been noted that the corrosion of the plates of a steam boiler is largely increased at points where there is concentration of stress or vibration; that is to say, along the riveted joints. The grooving is sometimes very marked indeed at such places, when the corrosion of the general surface of the plate is very slight.

Mr. Baylis. MR. JOHN R. BAYLIS.[3]—There is one thought of Mr. Speller's discussion that should perhaps be emphasized, and that is the calcium carbonate solubility equilibrium of the water in which the tests were made. I should like to suggest that a water corrosive to calcium carbonate be selected for one set of experiments. Sea water should be saturated with calcium carbonate and it is likely that brackish water is somewhere near the saturation point.

Calcium carbonate, when the concentration is fairly high, is quite effective in stopping the corrosion of ferrous metals, and apparently has considerable effect upon some of the alloys. Whether this would affect the results given by the author to any material extent, I do not

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[1] Speller and Texter, "Effect of Alkaline Solutions on the Corrosion of Steel," *Industrial and Engineering Chemistry*, Vol. 16, pp. 393–397 (1924).

[2] Engineer of Materials, New York Central Lines, New York City.

[3] Filtration Engineer, City of Chicago, Chicago, Ill.

Mr. Baylis.

know; unless a corrosive water is tried one will wonder what influence such a water would have. It would seem better if a water from some public water supply known to be corrosive were selected, something like the Catskill supply for New York City. For waters practically free from soluble salts except calcium carbonate, the saturation equilibrium of calcium carbonate is about 14 to 15 parts per million when the pH is slightly above 9.0. The lower the pH the more calcium carbonate there will have to be in solution to establish saturation equilibrium. With a pH of about 8.0 the concentration at the saturation point of calcium carbonate is about 50 parts per million, and at pH of 7.6 the saturation point is about 100 parts per million.

In the corrosion of ferrous metals, the first thing that happens when the metal goes into solution is to take some of the CO_2 out of the water. This throws the pH up and produces a supersaturation of calcium carbonate if it is fairly high. When the concentration is over 25 to 30 parts per million, calcium carbonate crystals will be formed. This helps to form an impervious membrane over the metal surface that greatly inhibits corrosion. I suggest in selecting waters for conducting the experiments that at least one very soft and corrosive water be tried. The concentration of calcium and magnesium carbonates or bicarbonates should be not over 15 parts per million.

Mr. H. F. Moore.

MR. H. F. MOORE.[1]—At the University of Illinois there have been carried out within the past year a few corrosion-fatigue tests, which, while they are not extensive enough to report as a special series, yet may be worth reporting as information. Specimens of heat-treated chromium-nickel steel were tested in reversed flexure under ordinary conditions and subjected to a stream of water. The water used was the ordinary University supply, coming from wells about 135 ft. deep—the same water supply which caused "caustic embrittlement" in certain boiler drums, as described in the papers by Parr and Straub.[2]

Fatigue specimens 0.3 in. in diameter showed a reduction of endurance limit by corrosion-fatigue of over $66\frac{2}{3}$ per cent (tests not yet finished). Fatigue specimens 1 in. in diameter showed a reduction of 45 per cent. While this result is only preliminary it indicates that size of specimen may have some effect on corrosion-fatigue.

Fatigue specimens, 0.3 in. in diameter, of cast iron No. 93, described in the paper presented this year by Mr. Lyon and the speaker,[3]

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[1] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] S. W. Parr and F. G. Straub, "The Cause and Prevention of Embrittlement of Boiler Plate," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 52 (1926); also, "Embrittlement of Boiler Plate," see p. 52.

[3] H. F. Moore and S. W. Lyon, "Tests of the Endurance of Gray Cast Iron Under Repeated Stress," see p. 87.

Mr. H. F. Moore. showed no reduction of endurance limit under the action of a stream of water.

Mr. McAdam. MR. McADAM (*author's closure*).—Mr. Lessells' discussion gives the impression that he disapproves of presentation of data on fatigue of metals unless the data are accompanied by a complete theory of the mechanism of fatigue. The facts and relationships in the present paper, however, are presented because of their practical interest. They are available for theoretical consideration by Mr. Lessells and others. My tentative theoretical discussion gives due weight to the various hypotheses without reaching a final conclusion. I believe that is as far as we can go at present.

I do not agree with Mr. Lessells' idea that it is better to focus attention on reduction of fatigue strength rather than to compare corrosion-fatigue limits and study the effects of variables on these limits. In the first approach to the subject of corrosion-fatigue it is natural to discuss it mainly from the point of view of reduction of fatigue strength. It was so discussed in the first paper by the author. Even at that time, however, the idea of intrinsic corrosion-fatigue limits was suggested. The idea of intrinsic corrosion fatigue limits has continually increased in practical and theoretical value.

Mr. Lessells' discussion of the effect of elastic limit on the corrosion-fatigue limit is essentially a paraphrasing of the discussion of this subject in the paper. The only addition that he makes is to mention an additional example taken from a previous paper.

His discussion of the effect of nickel is not clear. He merely cites a number of examples without explaining what they are intended to prove.

The idea may be gathered from Mr. Lessells' discussion that he considers the approximate identity of the corrosion-fatigue limit for cold-worked and for fully-annealed material to be due to a balancing of two opposing influences. His idea appears to be that the influence of increase in strength due to cold working is just balanced by the influence of a decrease in corrosion resistance. In discussing one of my previous papers Mr. N. L. Mochel advanced a similar idea to explain the relative smallness of variation of the corrosion-fatigue limit of carbon steels with variation in composition and heat-treatment. This hypothesis of Mr. Lessells, however, will not withstand close analysis.

Mr. Lessells refers to a paper by Goerens on the effect of cold working on electrolytic potential of steel. In this paper Goerens showed that the difference in potential between cold-worked and fully-annealed specimens may amount to several hundredths of a volt. In his use of such facts as a basis for his hypothesis, Mr. Lessells has overlooked several important points which will now be discussed. **Mr. McAdam.**

In considering corrosion-fatigue, it should be obvious that the important factor is not rapidity of general corrosion or the electrolytic potential of the specimen as a whole. The important factor is the electrolytic potential at points of maximum potential, such as slip lines, grain boundaries and inclusions. The corrosion-fatigue limit depends, not on the number of such points, but on their potential. The probable influence of potential at slip lines and grain boundaries is discussed briefly in the first complete paragraph on page 123 of the paper.

Mr. Lessells also fails to realize that in corrosion-fatigue the important factor is corrosion-resistance under cyclic stress. Under such conditions slip lines are being continually formed. They are formed in large quantity in annealed material, for which the corrosion-fatigue limit may be above the elastic limit or proof stress, and may even be above the endurance limit. For material HE–14 of Fig. 3, for example, the corrosion-fatigue limit is above the endurance limit. In a specimen of this material under test at the endurance limit there is so much hysteresis that the specimen becomes warm. Does it not seem probable that in such a specimen the electrolytic potential at the slip lines is at least equal to that in previously cold-worked material? And yet for this material the corrosion-fatigue limit is above the endurance limit, while for the cold-work material *HE*-4 the corrosion-fatigue limit is below the endurance limit. These and many other similar facts are not explained by Mr. Lessells' hypothesis.

Mr. Gillett makes a very commendable effort to induce others to go into the field of corrosion-fatigue testing. In this attempt, however, it seems possible that his discussion may cast doubt on some of the results obtained by the author. He refers to a paper by G. D. Lehmann[1] which was published last December. Mr. Lehmann[1] made corrosion-fatigue tests in the following strong solutions of salts: Sodium nitrate 1 part, water 2 parts; sodium chloride 1 part, water 3 parts; ammonium chloride 1 part, water 3 parts. He says that his object in making the solutions so strong was to minimize corrosion. He wished to see if these strong solutions, heated nearly to the boiling point, would cause embrittlement of steels due to absorption of nascent hydrogen. For this purpose he surrounded the specimen with a relatively small rubber tube containing only a small quantity of liquid in contact with the specimen. Air was excluded.

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[1] G. D. Lehmann, "The Variation in the Fatigue Strength of Metals when Tested in the Presence of Different Liquids," British Aeronautical Research Committee, Reports and Memoranda No. 1054, October, 1926.

Mr. McAdam. Mr. Gillett takes these experiments by G. D. Lehmann, in which he was using concentrated solutions with the idea of minimizing corrosion, and contrasts them with the results obtained at the Naval Experiment Station with dilute sea water and with easy access of air. This leads him to say that it is hard to reconcile the results obtained in the two laboratories. This example cited by Mr. Gillett, however, merely illustrates the fact that corrosion is a variable factor. It does not cast any doubt on the results presented by the author.

Mr. Gillett says that a few graphs in the paper were extrapolated beyond what the individual results seem to indicate. That is true, and it was referred to in the paper. These graphs were drawn by analogy. We now have enough corrosion-fatigue graphs, so that extrapolation can be used in certain conditions when we have not enough specimens to make a complete graph. Extrapolation is used now with ordinary fatigue graphs when we have few specimens. This is a proper procedure in view of the large number of fatigue tests that have been made and published.

Mr. McKay points out that corrosion-fatigue limits cannot be used directly in design. It is true that great care must be used in attempting to use corrosion-fatigue limits in deciding on dimensions of machinery parts. As the corrosion-fatigue limit varies with conditions of corrosion, the dimensions of a machinery part should depend somewhat on the severity of the corrosion.

The corrosion-fatigue limits published in the paper, however, give an idea of the relative value of various alloys under corrosion conditions kept as nearly constant as possible. It is hard to avoid variation in corrosion conditions in our corrosion-fatigue experiments. If the water stream does not surround the specimen uniformly, corrosion is more severe in regions where the stream is thinnest, and in that region the specimen is likely to break. So it is rather surprising that we do not get more erratic results because of the difficulty of keeping the corrosion conditions constant.

Corrosion-fatigue limits obtained in this way are of value in determining the relative value of various alloys when subjected to fatigue under specific conditions of corrosion. As a guide to selection of material, which is an important part of design, corrosion-fatigue limits should be of great value.

Mr. McKay refers to the effect of varying cycle frequency, which results in varying corrosion time per cycle. This subject was discussed in a previous paper.[1] We are continuing tests at low cycle

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[1] D. J. McAdam Jr., "Corrosion-Fatigue of Metals as Affected by Chemical Composition, Heat Treatment, Cold Working, Part I," *Transactions*, Am. Soc. Steel Treating, March, 1927, Vol. XI, No. 3.

Mr. McAdam. frequency, 50 r.p.m. and 4 to 8 r.p.m. As such tests require 28 to several hundred times as long as the tests at 1450 r.p.m. reported in the paper, progress is slow. Interrelationship between the effects of time, cycle frequency, and stress is being investigated. The results will be presented in a later paper.

Mr. McKay is inclined to attribute the relative constancy of the corrosion-fatigue limit for nickel-copper alloys to the presence of a second phase, such as magnesium sulfide. It does not seem probable, however, that the presence of small scattered particles of magnesium sulfide would have more effect than non-metallic inclusions similarly distributed. The corrosion-fatigue limit probably depends chiefly on the properties of the predominant phase. Investigation of a great variety of metals and alloys has shown that the corrosion-fatigue limit apparently depends largely on the electrochemical properties, rather than on physical properties. This is illustrated not only by nickel-copper alloys but by copper-zinc alloys and by other alloys to be discussed in a later paper. Possible reasons for the apparent lack of influence of physical properties have been discussed in this paper and in preceding papers.[1,2]

Mr. R. R. Moore. MR. R. R. MOORE (*by letter*).—Mr. McAdam has uncovered a very interesting phenomenon in the tests on the annealed 21:78 nickel-copper alloy where the corrosion-fatigue limit was found to be higher than the fatigue limit. Annealed material is generally more susceptible to strain hardening than is hardened material. It is suggested that the strain hardening produced by repeated stressing may have some effect upon the progression of the corrosion. Specimens tested after preliminary corrosion might develop valuable information along these lines for the corrosion would have already taken place.

The constancy of the corrosion-fatigue limits of the nickel-copper alloys is very interesting. Mr. McAdam's suggestion that this may be due to a second phase, of nearly constant composition for a large range of nickel-copper alloys, in the grain boundaries is quite logical. The intercrystallin corrosion of duralumin is now an accepted fact. Furthermore, there is good reason to believe that this corrosion is due to the presence of $CuAl_2$ particles in the grain boundaries. It is further suggested that the wide scatter of test results, and the long runs required to locate the endurance limit for duralumin as has been reported by both Mr. McAdam and the writer, may be due to the

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[1] D. J. McAdam, Jr., "Stress-Strain-Cycle Relationship and Corrosion-Fatigue of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 224 (1926).

[2] D. J. McAdam, Jr., "Corrosion-Fatigue of Metals as Affected by Chemical Composition, Heat Treatment, Cold Working, Part I," *Transactions*, Am. Soc. Steel Treating, March, 1927, Vol. XI, No. 3.

Mr. R. R. Moore. progressive intercrystallin corrosion of that alloy even under conditions in which duralumin gives no external evidence of corrosion. The writer has found intercrystallin corrosion in duralumin taken from airplanes, even though the surface of the material was apparently unattacked.

Mr. McAdam. MR. MCADAM (*author's closure by letter*).—The influence of dissolved calcium carbonate in the fresh and salt water used in this investigation has been discussed by Mr. Baylis. It becomes increasingly evident that this calcium carbonate has great influence on corrosion-fatigue, especially corrosion-fatigue of steels. As shown in the paper the fresh and salt-water corrosion-fatigue limits for a non-ferrous metal do not differ greatly. For steel, however, the salt-water corrosion-fatigue limit is considerably below the fresh-water limit. It seems probable that this relatively great difference is due to heavy precipitation of calcium carbonate from the fresh water. It is planned to make some corrosion-fatigue tests with soft water as Mr. Baylis suggests.

In this paper and previous papers it has been emphasized that the corrosion-fatigue limit apparently depends more on the electrochemical than on the physical properties. It has also been repeatedly pointed out that the apparent lack of influence of physical properties may be due to a more or less complete balance between two effects of a physical property. Increasing hardness, for example, may be accompanied by increasing stress concentration in small corrosion pits. That such a balance of effects of physical properties is the cause of the apparent lack of influence of physical properties on the corrosion-fatigue limit appears more probable as a result of recent experiments to be described in a later paper. The important fact remains, however, that for specimens whose diameter is ½ in. or less the corrosion-fatigue limit depends largely on the electrochemical properties. With increase in the size of the specimen it seems probable that the influence of strength will become more prominent and the influence of electrochemical properties will become less prominent. Definite conclusions on effect of size, however, must await the results of experiment.

FATIGUE STUDIES OF TELEPHONE CABLE SHEATH ALLOYS

By John R. Townsend[1]

Synopsis

This paper describes fatigue studies of lead sheath for telephone cables, the development of two forms of simulated service test and a fatigue machine designed to test lead and its alloys. The correlation between the characteristics of service failures and the laboratory failures is stressed. The special precautions necessary in carrying out fatigue tests on soft metals is outlined.

Fatigue failure of lead and the lead-antimony alloys covered by this paper is by intergranular failure. In the case of the lead-antimony alloys repeated stress appears to reduce the solid solubility of antimony in lead, producing a widened grain boundary as viewed under the microscope.

The type of fatigue test described is sensitive to very slight changes in composition and heat treatment for a particular class of materials. For example, the effect of impurities in the base metal and slight changes in composition is strikingly brought out by the results of the fatigue test.

Introduction

Lead-covered aerial telephone cable is being used extensively to replace open-wire construction wherever feasible, particularly in sections of the country subject to sleet storms. Such cable is suspended from a galvanized steel wire strand by means of rings spaced about 16 in. apart, except at poles, where the spacing is reduced.

A full-size telephone cable has an over-all diameter of $2\frac{5}{8}$ in., a lead-antimony sheath $\frac{1}{8}$ in. thick and weighs about $7\frac{1}{2}$ lb. per ft. The steel wire strand has a breaking strength of over 16,000 lb.

Aerial cables are subject in service to varying stresses due to several causes, such as wind swaying the cable, road shocks transmitted through the supporting structures, and temperature changes, daily and seasonal. It is evident that the most of these causes would introduce stresses which would vary in magnitude as well as in rate of change. It would seem reasonable, therefore, to conclude that fractures which develop in service in the lead sheath near the poles are due to fatigue under the action of repeated stresses.

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[1] Bell Telephone Laboratories, Inc., New York City.

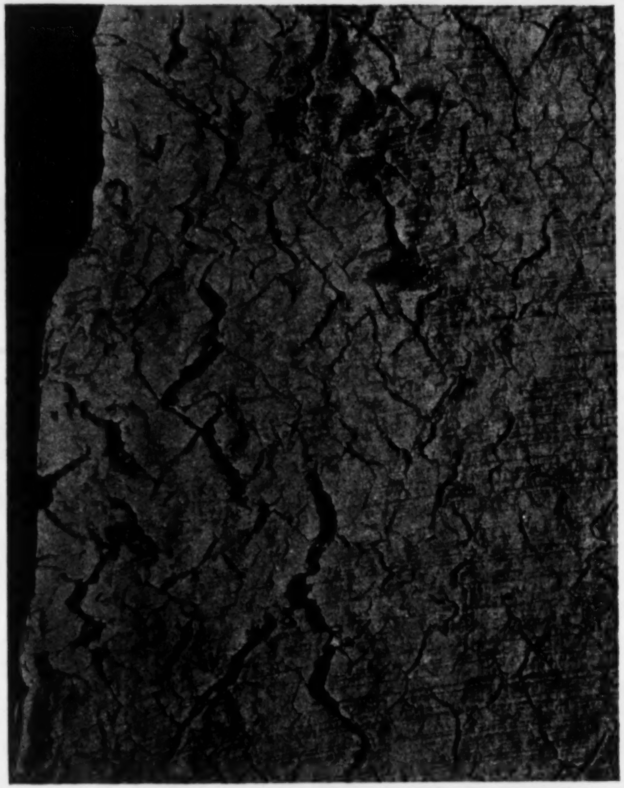

FIG. 1.—Showing Typical Failure With Pure Lead Sheath and Extent of Intergranular Failure; Unetched. (× 3½.)

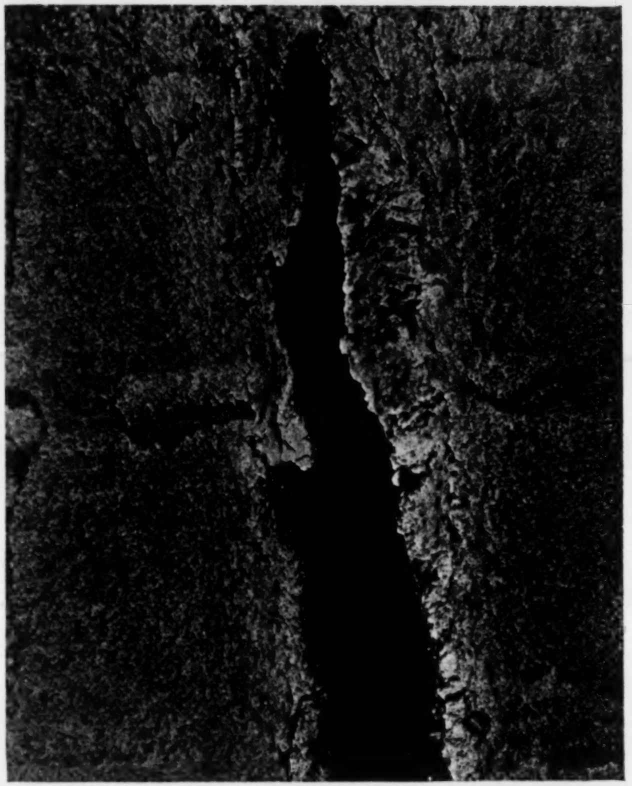

FIG. 2.—Showing Typical Lead-Antimony Cable Sheath Failure; Unetched. (× 3½.)

However, an investigation was made of the possibility of these service failures being due to sustained stress resulting from slow changes in length caused by seasonal changes in temperature. The alloy most used for telephone cable sheath consists of 99 per cent lead and 1 per cent antimony. Tension test specimens were cut from full-size cable sheaths. One end was clamped and to the other end was attached a load sufficient to produce in the specimen a stress of 1350 lb. per sq. in. This stress approximates the proportional limit for this material when stretched at a rate of 0.03 in. per minute of the traveling head of the testing machine in standard tension tests.

At this stress the lead appears to stretch continuously at a rate which is of the order of 0.0002 in. per inch of length per minute. Fracture finally occurs after over 800 hours, with a total elongation of about 36 per cent. This is about the elongation obtained in a standard tension test specimen where the load is rapidly applied. However, there is none of the necking down of the material at the point of fracture which is characteristic of a standard tension test.

Photomicrographs showed that failure occurred by separation of the crystal grains transversely to the direction of the stress, thus indicating a flow or movement of the grains. This condition is not found in service fractures. Furthermore, it is self-evident that any such elongation of the sheath could not take place in service because of the presence of the core. It is, therefore, concluded that service failures are due to repeated stress. This is further confirmed by an analytical study of the actual conditions existing at the points where failures occur, namely, near the poles. Figure 1 shows a typical failure of the pure lead sheath originally used, and Fig. 2 a lead-antimony sheath failure. In Fig. 1 the failure shows the brittle intergranular type of breakage, whereas in Fig. 2 the break is more localized as the alloy has greater fatigue resistance.

Based on this study of the conditions existing in service where failures occur, two types of testing machines were first developed to simulate those conditions, namely, a "vibration machine" and a "bending machine." A series of tests was devised which would permit improvements in the design of the cable and in the composition of the sheath. These machines employed specimens of cable, but later a fatigue testing machine was developed which employed specimens of the sheath.

The purpose of this paper is to describe briefly these machines and some of the results obtained in the various investigations.

VIBRATION AND BENDING TESTS

Vibration Machine.—The vibration machine is shown in Fig. 3. Specimens of full-size cable with core are clamped at one end, and the other end is vibrated transversely. The cable specimen is approximately 40 in. long and the free or vibrated length, not included in the clamps, is 24 in. The specimens are vibrated at the speed of 1700 r.p.m. and the amount of displacement of the movable head is approximately $\frac{1}{8}$ in. at the center of the clamps. This machine has a capacity of eight specimens, four on each side of the vibrating mechanism. The specimens are clamped between wood blocks. At the stationary clamps they are supported internally by a round steel wedge that is forced into the end of the cable for the length of the clamps, pushing aside the conductors, thus tending to prevent crushing of the cable in the clamps. The specimens of cable are sealed at each end and a slight vacuum is produced within them. At the stationary end they are attached to a mercury manometer. Failure of the sheath permits air to enter and destroy the vacuum, causing the manometer to register a failure through an electrical connection.

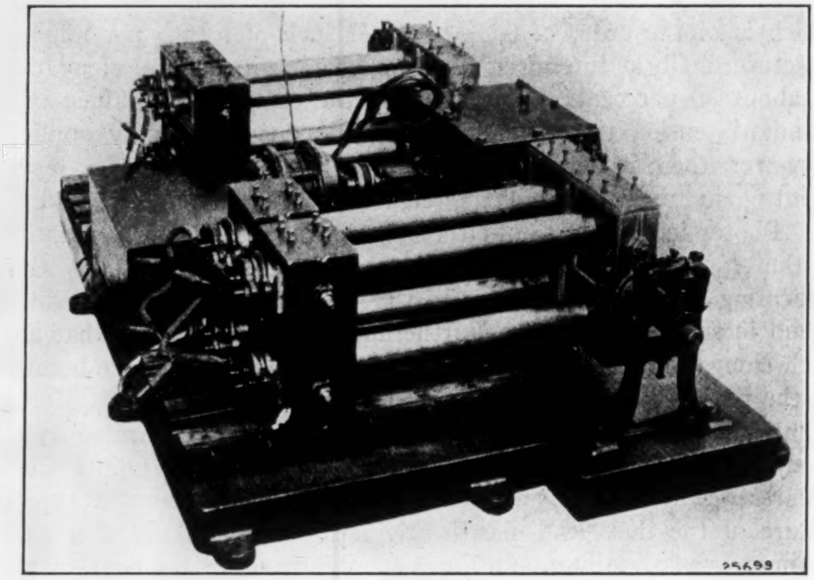

FIG. 3.—Vibration Machine.

Bending Machine.—The bending machine is shown in Fig. 4. The specimens consist of full-size standard cable 9 ft. long. The specimens are bent into the form of a U, having a radius of 18 in. One end of the specimens is clamped stationary and the other end is attached to a movable head. This movable head travels back and forward, a distance of 2 in., at a constant speed of approximately 96 bends per minute. The cable is carefully bent into the U shape by a fixture so that no kinks or buckles can occur in the specimens.

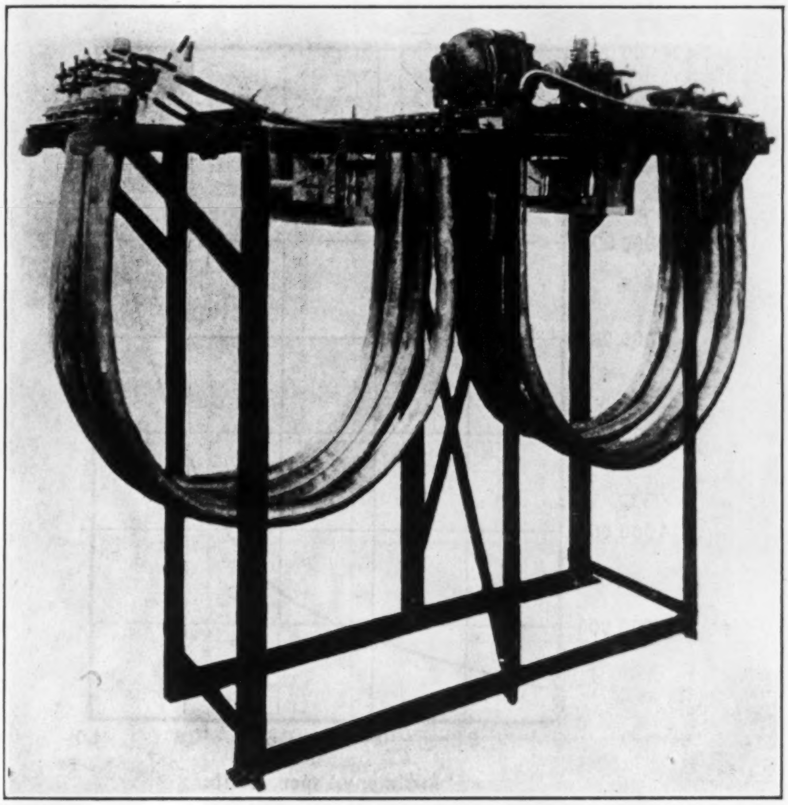

FIG. 4.—Bending Machine.

The capacity of the machine is eight specimens and the failure is recorded in the same manner as described for the vibration machine.

Results of Vibration and Bending Tests.—The results of typical runs on the vibration and bending machines are shown in Figs. 5 and 6. These curves illustrate strikingly the effect of antimony on the fatigue resistance of cable sheath. Results from these tests are convincing in that they are consistent with the behavior of cable under service conditions but are exaggerated to the point where failure occurs in a reasonable length of time.

FATIGUE TESTS OF SHEATH SPECIMENS

Whereas the tests just described reveal the fatigue-resisting qualities of cable, it was considered advisable to develop a test that would employ the sheath alloys alone and eliminate the unavoidable variables that affect the results of a test employing cable as specimens. Furthermore, it was seen that if a satisfactory small specimen could be developed, results could be obtained on a great number of specimens in a comparatively short time. It is recognized that fatigue testing in general is not a precise art, and it is advisable to test a large number of specimens to obtain sufficient data upon which to base reliable conclusions.

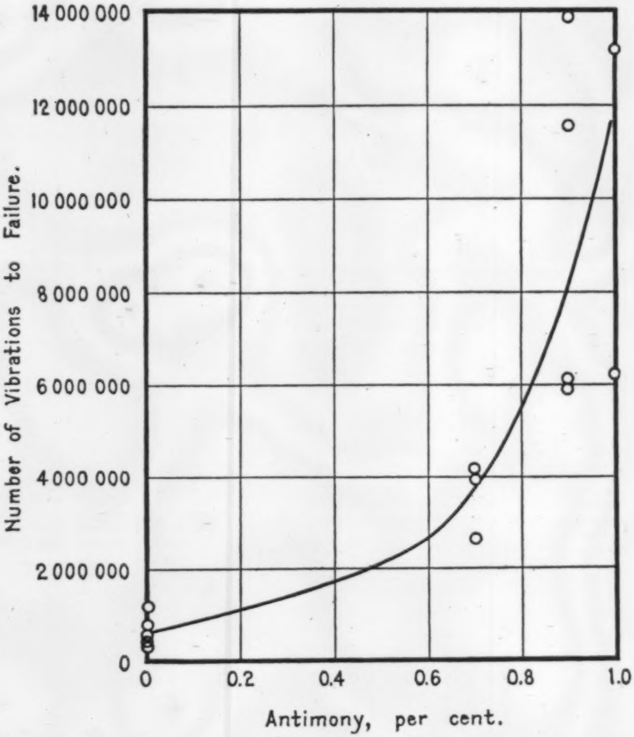

FIG. 5.—Vibration Tests on Full-Size Lead-Antimony Cable Sheath.

In developing the fatigue test described below, it was felt that the form of the test should not depart radically from the conditions of service; in other words, the action of the machine should resemble in kind, if exaggerated in degree, the action of the cable in service. While the use of a round beam test piece and the determination of an endurance limit by means which are more or less familiar to testing engineers, had much to recommend them, considerable time would be required to obtain the results, and in view of the peculiar properties of lead, considerable experimental work would be required to adapt existing testing machines to the work at hand. Furthermore, even if the endurance limits for the various materials could be determined, there would be considerable difficulty in applying the results of these tests to the engineering of cable structures.

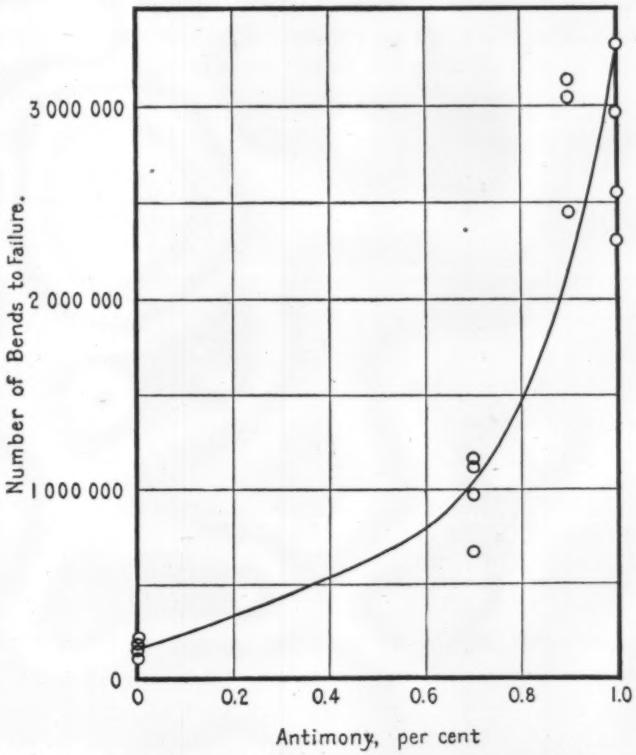

FIG. 6.—Bending Tests on Full-Size Lead-Antimony Cable Sheath.

It was considered desirable, therefore, to lay aside the determinations of endurance limits of lead sheath and instead to determine the *relative* resistance of various proposed materials to repeated stress conditions, exaggerated perhaps, but not unlike the conditions which these materials would have to withstand in service. By this means, fairly reliable circumstantial evidence could be obtained. The number of repetitions of stress to produce failure is described hereinafter as the "fatigue resistance" of the particular material under investigation.

Cable is stressed in service in one direction only and then returns towards zero stress, or in other words cable is bent and vibrated mostly in one direction and does not alternate between tension and compression as would be the case with an alternately stressed beam. It was decided, therefore, to stress the specimens in one direction only. By so doing, a very simple form of cam-type machine could be devised.

Fatigue Test Specimens.—The specimens were cut from extruded cable sheath by slitting the sheath and flattening it carefully in a press. Considerable data were obtained with these specimens, but it was considered that better agreement in the test results could be obtained by extruding material in the form of flat tape, thus avoiding variations in thickness of the specimens due to eccentricity in the sheath and the possibility of injuring the material by flattening it before the specimens could be prepared. This proved to be the case, and except where it has been necessary to test actual sheath all of the material under investigation has been in the form of extruded tape. The tape was $1\frac{1}{2}$ in. wide and $\frac{1}{8}$ in. thick. The extrusion pressure and the temperature of the die blocks were carefully controlled and maintained at an average of commercial extrusion conditions. The material was carefully handled in coming from the press so that no bends or bending stresses were introduced. Fatigue specimens and tension specimens were alternated along the length of the tape.

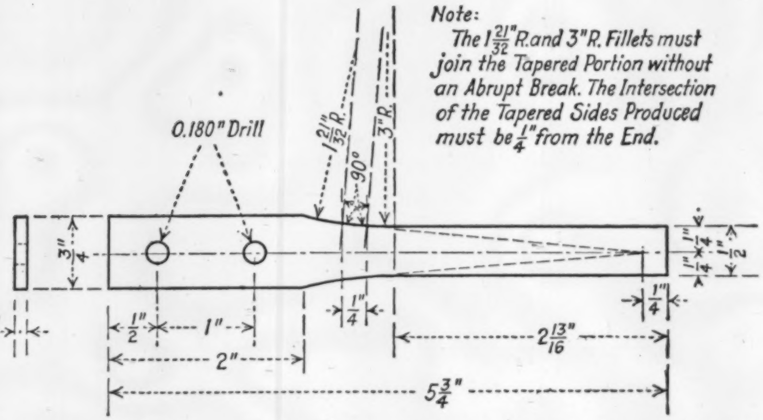
Note:
The $1\frac{21}{32}$" R. and 3" R. Fillets must join the Tapered Portion without an Abrupt Break. The Intersection of the Tapered Sides Produced must be $\frac{1}{4}$" from the End.
0.180" Drill
$1\frac{21}{32}$" R.
3" R.
90°
$\frac{3}{4}$"
$\frac{1}{8}$"
$\frac{1}{2}$"
1"
2"
$\frac{1}{4}$"
$2\frac{13}{16}$"
$5\frac{3}{4}$"
$\frac{1}{4}$"
$\frac{1}{4}$"
$\frac{1}{4}$"
$\frac{1}{2}$"

FIG. 7.—Experimental Fatigue Test Cantilever Specimen for $\frac{1}{8}$-in. Lead Cable Sheath Alloy.

Figure 7 shows the cantilever beam specimen employed. It is so designed that it has a uniformly stressed length of $\frac{1}{4}$ in. The method of eliminating the effect of clamping stresses, which is an important consideration in a material of the character of lead, is to connect the uniformly stressed portion to the clamped section by means of a fillet. Before this final design of specimen was decided upon a number of different specimens of this form were tried using various uniformly stressed lengths. A specimen with a longer uniformly stressed length required a greater number of deflections on the fatigue machine to cause breakage. This was probably due to the greater length permitting the lead alloy to adjust itself to localized stresses in the material and thus resist a greater number of deflections. Conversely, a shorter uniformly stressed free length led to difficulties due to the uncertainty of preparing a specimen to sufficiently exact dimensions. The original specimens were prepared by milling, using a carefully prepared template. Later a special profile milling cutter was employed and a number of specimens could be cut simultaneously by cross-milling. This method was also employed in the preparation of the tension specimens.

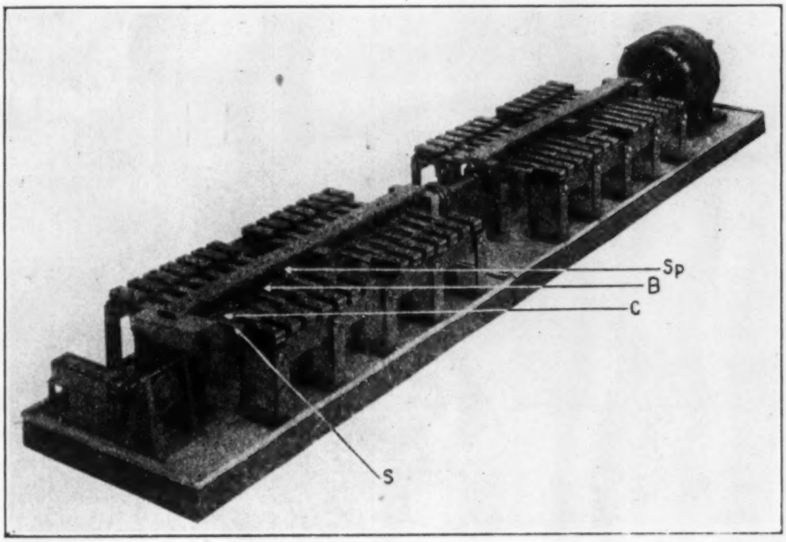

FIG. 8.—Fatigue Machine Employing Small Cantilever Specimens.

Fatigue Testing Machine.—The fatigue machine consists of a cam *C*, Fig. 8, which rotates at 700 r.p.m. This speed was found, after considerable experiment, to be the practical maximum. The specimen, *S*, is clamped at one end and the other end is in contact with the cam. In order to prevent wear between the cam and the specimen, a phosphor-bronze boot, *B*, is slipped over the end of the specimen. Whereas the specimens are stressed within their apparent elastic range, yet after a considerable number of deflections there occurred a slight drifting away from the cam. This resulted in pounding. Futhermore, irregularities, however slight, caused vibration that produced unknown stresses within the springs. These difficulties were taken care of by employing a low-tension phosphor-bronze compression spring *Sp* to hold the specimen against the cam. The machine consists of two units in tandem, accommodating in all 56 specimens. A $\frac{1}{8}$-h.p., d.c. shunt motor is used to drive the machine.

Considerable thought was given to the elastic properties of lead. If measurements of elongation are sufficiently refined and the time of loading long enough, it is possible that no perfectly elastic range could be determined for the 1-per-cent antimony-lead alloy. If a specimen of lead is deflected a given amount it will tend to return to its original position depending upon the time of application of the load, the temperature, and the amount of the deflection. What was desired in these tests was to be able to deflect the specimens of sheath material a sufficient amount to keep the stress below a value that would cause flow and to repeat the stresses at a rate that would produce breaks within the shortest possible time. Therefore, a speed was selected that appeared to be the practical maximum and a deflection that was within a stress value that would not cause plastic action of the material. This range of stress we have termed the "apparent elastic range" for the rate of operation and at a room temperature of 80 to 90° F.

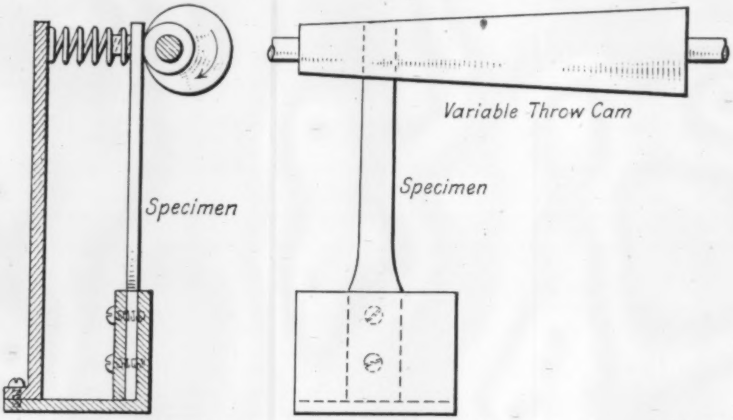

FIG. 9.—Schematic Representation of Machine Designed to Determine Apparent Elastic Range of Lead Cable Sheath Alloys.

The tensile strength varies with the rate of movement of the pulling head of the testing machine; for example, an increase in speed of 100 per cent produces an increase in tensile strength of 10 per cent. The following method, illustrated schematically in Fig. 9, was used eventually to determine the apparent elastic range: A variable throw cam was rotated at 700 r.p.m., the speed of the fatigue machine. The specimen was clamped in a vertical manner, so as to eliminate the effect of its own weight, and traversed from left to right by a screw and slide mechanism. In going from left to right, the end of the specimen bearing on the cam was caused to be deflected a uniformly increasing amount. In this instance, the boot used on the end of the specimen had a hemispherical bearing surface. This gave point contact and prevented the specimen from being twisted on the variable throw of the cam. The vibrating end of the specimen was backed up by the compression spring as used on the fatigue machine. Therefore, by traversing from left to right and then back to zero in small increments, eventually a point was reached where the specimens took a permanent set, found by removing the spring, and thus indicating that the material had been stressed beyond its apparent elastic range. In this way all the experimental alloys were tested in order to determine their apparent elastic ranges.

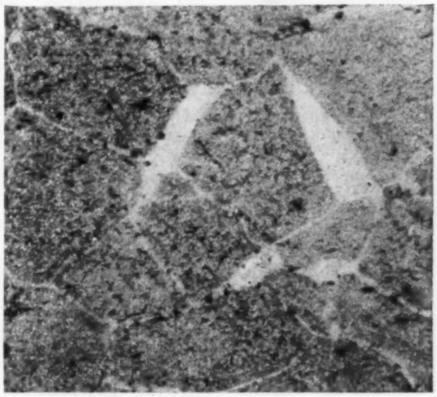

FIG. 10.—Photomicrograph of Lead Cable Sheath Alloy Adjacent to Service Failure. (×200.) Etching Reagents, 6 cc. Glacial Acetic Acid and 2 cc. Superoxol (30 per cent Hydrogen Peroxide), 6 to 15 seconds.

It was found that a deflection of 0.11 in. in a length of 3 in. corresponded to a calculated stress of 1350 lb. per sq. in., which was just within the apparent elastic range of most of the experimental alloys investigated. The "fatigue resistance" of the material is then the number of repeated bending stresses at this stress. In this manner all of the alloys could be compared and results obtained in a reasonable length of time. Should a material be found that had a fatigue resistance considerably in excess of the other materials under investigation, then a cam of a greater throw would be employed and this material compared directly at this stress with other materials in its same class. This procedure of testing has much to recommend it in carrying out simple comparisons and especially for detecting the effects of small amounts of constituents added to a given class of alloys.

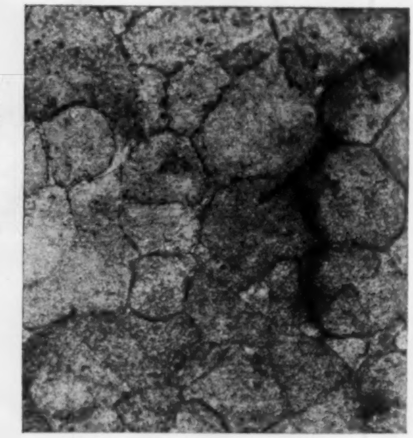

FIG. 11.—Fatigue Specimen Sectioned Through Maximum Stressed Area. (×120.) Etching Reagent, 6 cc. Glacial Acetic Acid and 2 cc. Superoxol (30 per cent Hydrogen Peroxide), 6 to 15 seconds.

The cracks proceed from the outside surface of the specimen toward the the center. Note the progressive widening of the grain boundaries.

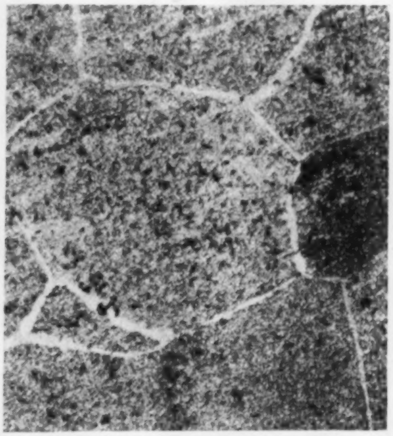

FIG. 12.—Condition of Lead-Antimony Alloy After Nine Years Service. (×200.) Etching Reagent, 6 cc. Glacial Acetic Acid and 2 cc. Superoxol (30 per cent Hydrogen Peroxide), 6 to 15 seconds.

Results of Fatigue Specimen Tests.—If the fatigue specimens were stressed beyond their apparent elastic range, the breaks would be similar to a tension break. Within the apparent elastic range, however, the breaks compared in appearance with the breaks that occur in service. Figure 10 shows a photomicrograph of a section adjacent to a service failure. Figure 11 shows a fatigue specimen sectioned through the maximum stressed area after 3,000,000 deflections. Note the intergranular type of failure in each case.

Further evidence of the similarity of service with laboratory failure is in the behavior of the lead-antimony alloy. Figure 12 shows a section of lead-antimony near to a pole which has been in service nine years. Note the widening of the grain boundaries. Referring again to Fig. 11, showing a fatigue specimen after 3,000,000 deflections, the progressive widening from the center or neutral axis of the cantilever specimen toward the outside or maximum stressed area will be noted. This shows that stress is largely responsible for the widened condition of the grain boundaries, which indicates a change in the amount of antimony remaining in solid solution in lead, a phenomenon first pointed out for this alloy by Mr. Francis F. Lucas who also prepared the photomicrographs shown in this paper by methods described elsewhere.[1]

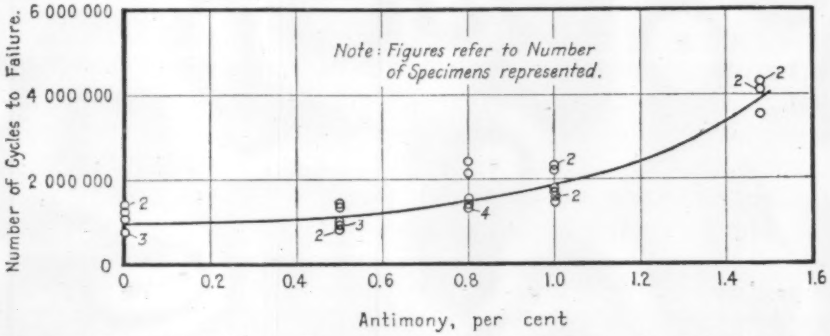

FIG. 13.—Effect of Antimony Content on Fatigue Resistance of Lead Cable Sheath Alloy.

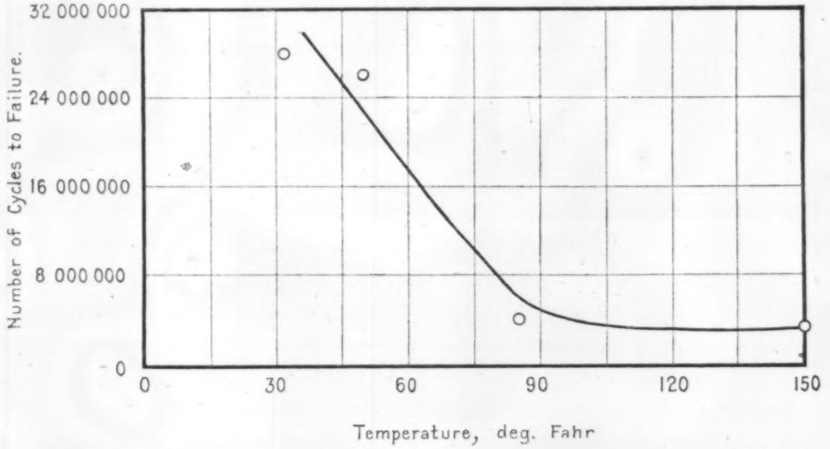

FIG. 14.—Curve Showing Effect of Temperature on Fatigue Resistance of Lead Cable Sheath Alloys.

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[1] F. F. Lucas, "Application of Microtome Methods to the Preparation of Soft Metals for Microscopic Examination," Pamphlet No. 1654-E issued with *Mining and Metallurgy*, February, 1927.

Typical results of the fatigue tests using extruded tape are given in Fig. 13. These results should be compared with the results of the vibration and bending tests shown in Figs. 5 and 6. Note how sensitive the fatigue test is to slight changes in composition of the alloy. Also note the lack of dispersion of the results on the fatigue tests.

Conclusions in regard to recommendation of the use of cable sheath alloys are based only on concurrent results of all of the forms of test, namely, the vibration test, the bending test and the fatigue test. The fatigue test is economical in presenting a careful preliminary survey of experimental sheath alloys and those alloys which show promise are then checked on the vibration and bending machines in the form of sections of commercial cable.

The fatigue test is very sensitive to changes in composition, heat treatment and the temperature and pressure of extrusion. This test is also sensitive to changes in room temperature. A change in room temperature of 20° F. exerts a noticeable influence upon the test results, as may be seen from Fig. 14. As might be expected, an increase in temperature weakens and a reduction in temperature strengtens the alloy in regard to its resistance to fatigue. For these experiments a small portable machine was used that could be placed in an ice box or Friez oven.

CONCLUSIONS

A vibration and bending machine are described which are based on the performance of aerial telephone cable in service.

A fatigue machine and specimen are described, designed to test a large number of cable sheath alloys in a short time. These alloys are compared in order to determine their resistance to a given fatiguing action. In this manner test conditions are created which are sensitive to slight changes in composition, heat treatment and structure of the alloys under consideration.

The similarity of service and laboratory breaks is illustrated.

The fatigue failure of lead and some of its alloys consists of an intergranular break.

Precipitation and coalescence of antimony from solid solution in lead are accelerated by strain.

Failure of the 1-per-cent antimony-lead alloy under long continued load is by separation of the crystal grains at their boundaries.

Acknowledgments.—The author is indebted to Mr. L. J. Burns who carried on practically all of the routine tests covered by this paper.

DISCUSSION

Mr. H. F. Moore.

MR. H. F. MOORE.[1]—So far as I am aware, this paper presents the first recorded study of the fatigue strength of a very soft, ductile metal, lead. I feel that Mr. Townsend is to be congratulated on his important contribution to our knowledge of the fatigue of metals.

I wish to call attention to the form of testing machine which Mr. Townsend has used for fatigue tests of lead. In both the machines for large specimens and the machine for small specimens the specimen is subjected to recurring cycles of a constant deflection. This type of machine is called by Gough, of the British National Physical Laboratory, a specific strain machine. Recurring cycles of constant deflection are what happen to a cable sheath in practice, rather than recurring cycles of a definite stress. In practice the sheath would not be called on to carry much load, but would be deformed following the curve of the cable, and if a cable swayed in the wind, the sheath would be subjected to repeated deflection.

Another good feature of the machine used by Mr. Townsend for small specimens (Fig. 9) is the variable throw of the cams, allowing variation in the deflection.

In interpreting his test results Mr. Townsend has chosen some definite deflection and has judged the merit of a given lead by the number of deflections a specimen will withstand before failure. In the study of the fatigue strength of harder metals this method has not proved satisfactory, and the testing of several specimens at various stresses (in this case at various deflections), the plotting of a stress-cycle diagram (in this case a deflection-cycle diagram) and the determination of a fatigue limit below which the metal will, apparently, have an indefinitely large endurance, and which limit is located at the stress (in this case deflection) for which the stress-cycle diagram becomes horizontal, has been found a more satisfactory method of evaluating test results. If metals are evaluated on the basis of fatigue limit they are not always arranged in the order of merit given by tests carried out as Mr. Townsend describes. Taking a specific instance, Fig. 6 of Mr. Townsend's paper shows that in the cable sheath with 0.7 per cent of antimony about a million deflections were necessary to cause fracture; for the cable sheath with 1 per

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[1] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

Mr. H. F. Moore.

cent of antimony about 3,500,000 deflections were necessary to cause failure. Mr. Townsend concludes that this proves the superior endurance value of the cable sheath with 1 per cent of antimony. However, judging by fatigue tests of other metals, the speaker would feel that while this conclusion has a high degree of probability, Mr. Townsend's test results had not definitely shown that cable sheath with 1 per cent of antimony would have a higher fatigue limit (expressed in terms of stress or of deflection) than would cable sheath with 0.7 per cent of antimony. Suppose that for the two metals we have deflection-cycle diagrams, and that at about 4,000,000 cycles of stress the diagrams cross. Then for higher values of cycles Mr. Townsend's rating would be reversed. Of course, the probabilities are against such crossing, but since Mr. Townsend's machine allows a variation in deflection, the speaker hopes that he will make some study of the complete deflection-cycle diagrams for various leads, and not confine his tests to a comparison between arbitrary points on the diagrams.

Again the speaker wishes to emphasize the importance of Mr. Townsend's contribution, and to note that the above criticism is a criticism of one point only, and not of the entire paper.

Mr. Rawdon.

MR. H. S. RAWDON[1] (*by letter*).—Intercrystallin fracture in a metal may arise in a number of ways, but its formation simply as a result of the stressing of a material is not at all common. Usually this type of failure indicates either a peculiar combination of conditions to which the metal is subjected such as the simultaneous application of stress and corrosion, or some unusual structural condition, usually an undesirable one, existing in the material. It is the latter which the author has emphasized in this report.

The author has called attention to a peculiar grain-boundary condition which he has described as "a widening of the grain boundary" and has interpreted this as indicating a change in the solid-solubility of antimony in lead resulting from the stressing of the metal. The evidence upon which this very important conclusion has been based has not been given either in this paper or the one referred to by the author's associate (Mr. Lucas) which relates to the structural effect of aging rather than stress application. The prospect of a change of this kind in the structural condition of an alloy at ordinary temperatures resulting simply from stresses of the magnitude ordinarily encountered in the regular service is, indeed, novel. If such a change can occur in lead, it is not at all unlikely that it can occur in other metals under somewhat analogous conditions, and

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[1] Physicist (Metallography), U. S. Bureau of Standards, Washington, D. C.

Mr. Rawdon. one is led to all sorts of fascinating speculations concerning the possible influence of such a condition on the "life" of metals under certain service conditions. In particular, it would seem that this condition should obtain in metals when stressed at elevated temperatures and hence play a decidedly important part in determining their usefulness for high-temperature service.

The writer would suggest that the author present more completely the data upon which he has based the conclusion relating to the effect of stress on the solid solubility of antimony in lead, either in discussion of the present paper or in a separate paper at a later date, for it must be admitted that the examination of a lead-antimony alloy after nine years' of service (Fig. 12) alone tells one nothing as to any change assumed to have occurred during the nine years' interval. Nor in the micrograph of the specimen which had been fractured by fatigue stresses (Fig. 11), does there appear to the writer to be any convincing evidence of such a change which would warrant the conclusion "that stress is largely responsible for the widened condition of the grain boundaries."

Mr. Whittemore. MR. H. L. WHITTEMORE[1] (*by letter*).—This paper shows what can be accomplished by a bold materials engineer who uses the available information on fatigue but does not let it hamper him in attaining his objective by the shortest possible route. Many of us would have worried a great deal if we had been working on this problem. We would have attempted to determine the "endurance limit" which, possibly, is not an important property of this lead sheath, and we would have experienced a great deal of difficulty in designing a fatigue machine which would apply known stresses to the specimen. The agreement which Mr. Townsend finds between the results of his tests and the life of the cable in service are convincing proof that he has made his investigation "pay."

The value of his paper, however, from the standpoint of the materials engineer interested in the general application of fatigue tests would have been increased had he included a summary of the service records upon which this conclusion is based.

As all his fatigue testing machines subject the specimens to bending stresses it is not evident why one is called the "vibration machine," another the "bending machine," and the last one the "fatigue machine." Undoubtedly the bending machine closely simulated service conditions and was very useful in estimating the value of the other tests.

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[1] Chief, Engineering Mechanics Section, VI-5, U. S. Bureau of Standards, Washington, D. C.; discussion published by permission of the Director of the U. S. Bureau of Standards, Washington, D. C.

Mr. Whittemore. In most of the graphs showing fatigue data, we find the scale for the number of repetitions of stress plotted horizontally. The fact that in this paper this scale is plotted vertically makes one wish for standardization which would assist the reader to grasp the essential facts easily.

The fact that service records agree with the results of these tests, causing deformations which are much greater than those which occur in service, is considered satisfactory evidence that these accelerated fatigue tests are reliable. For engineering metals used for load-carrying members, it has, in general, been found that if the stresses in the fatigue test are greater than the stresses which occur in service the test results are not reliable in judging the material. Comparing two materials *A* and *B* under stresses twice the working stress, *A* may be found much superior to *B*, but if the fatigue test is made, using the actual working stress, *B* may prove to be much the better material. It should be noted, therefore, that for no other material for which we have fatigue data do the results of accelerated fatigue tests make it unnecessary to make fatigue tests closely approximating service conditions. This fact should make us accept the results of any accelerated fatigue test with great caution.

Several years ago, for testing the ductility of sheet duralumin, we built a repeated bend machine for bending the specimen around a cylinder in one direction, then unwrapping it and bending it around a cylinder of the same diameter in the opposite direction. This cycle was repeated until the specimen failed. It would be interesting to test this cable sheath in this repeated bending machine in which the specimen must conform to the cylinders and compare the results with those given in this paper.

Engineers interested in advancing our knowledge of the fatigue phenomena of metals will hardly be satisfied until these lead alloys are tested by repeated applications of known stresses and their properties compared with those of the stronger ferrous and non-ferrous alloys, although when they are used to cover cables, the amount of deformation is determined, as it is in the case of house paint, by the structure which supports them.

Mr. R. R. Moore. MR. R. R. MOORE[1] (*by letter*).—Mr. Townsend has made a very valuable contribution in demonstrating that the microstructural character of service failures in lead antimony alloys are similar to failures obtained in the laboratory on fatigue machines.

On page 164, the statement is made that specimens stressed beyond

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[1] Chief, Physical Testing Branch, U. S. Air Corps, McCook Field, Dayton, Ohio.

Mr. R. R. Moore.

the apparent elastic range give breaks similar to a tension break, and those stressed within the elastic range give breaks similar to service failures. Can this be interpreted to mean that the former were found to be failures through the grains and the latter failures through grain boundaries? If this is so, then in running sufficient tests to establish an endurance curve, some of which tests might be above the elastic range and some below, both types of fracture would be found. It is suggested that such a condition might have considerable to do with the reversal of curvature that is sometimes found in endurance curves if it could be shown that the particular alloys developed types of failure similar to the lead-antimony alloys.

In regard to bend tests, I believe it would be most valuable to record the type of fracture found here. This would give an idea as to whether this short-time test really approaches service conditions.

Mr. Townsend.

MR. J. R. TOWNSEND[1] (*author's closure by letter*).—The author is glad that Mr. Moore has emphasized the method of interpretation of the fatigue results in terms of service. The philosophy upon which these tests were based as stated in the paper is that if various cable sheath materials are strained repeatedly at a value representing the maximum which can occur in service the materials are correctly evaluated for that service and further, this form of test lends itself to direct interpretation in terms of use. The endurance limit of cable sheath alloys has been determined and will be reported in the future.

The limitations of space prevented a complete review of the phenomenon of the widening of the grain boundary area which is characteristic of the 1-per-cent antimony lead alloy when subject to cold working and aging commented upon by Mr. Rawdon. This phenomenon has been described in several papers by associates of the author and was mentioned in the present paper as evidence of the reproduction of service failures by the laboratory tests.

The situation surrounding the solid solubility of antimony in lead is briefly as follows. It had been believed for many years that antimony did not form solid solutions with lead. In 1923, Dean[2] showed the existence of appreciable solid solubility of antimony in lead at the eutectic temperature. Shortly thereafter Dean, Hudson and Folger[3] showed that dispersion hardening takes place in these alloys. A further detailed study using the conductivity method was made by Dean, Zicknick and Nix.[4] They were unable to obtain

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[1] General Development Laboratory, Bell Telephone Laboratories, Inc., New York City.

[2] Dean, *Journal*, Am. Chemical Soc., Vol. 45, 1683 (1923).

[3] Dean, Hudson and Folger, ***Industrial and Engineering Chemistry***, Vol. 17, p. 1246 (1925).

[4] Dean, Zicknick and Nix, ***Transactions***, Am. Inst. Mining and Metallurgical Engrs., Vol. 73, p. 505 (1926).

Mr. Townsend. equilibrium values at the lower temperatures due to the slow rate of precipitation of the solute. Mr. Lucas'[1] paper mentioned above demonstrates by a series of photomicrographs that cold work and temperature causes profound structural changes in these alloys. The structure of freshly extruded sheath (2 hours old) is shown with slight traces of antimony in the grains or grain boundary areas. After holding at 100° C. for varying periods and also by aging at room temperatures for long periods the antimony was thrown out of apparent solid solution in solid black surface masses which were not removed by washing. Fatiguing in service or in the laboratory exaggerates these structural effects, producing widened grain boundary areas which is lead antimony solid solution and the excess antimony precipitated from apparent solid solution agglomerates and tends to collect at the center of the grains. A paper by Schumacker and Nix[2] shows a careful determination of the solidus line of the lead-antimony system. A further and more recent paper by Schumacker and Bouton[3] utilizing the work of Lucas gives the solid solubility of antimony in lead as determined by conductivity measurements on cold-worked alloys. In other words cold working (fatigue) causes precipitation of the excess antimony from the super-saturated solution. An equilibrium is reached by cold working in a few hours which might take years to reach under normal conditions. The lead-antimony solid solution at the grain boundary areas appears to be weaker than the surrounding material and the breaks naturally occur here.

Both Mr. Whittemore and Mr. R. R. Moore stress the correlations of test results with service results. The author feels that this is very important and our testing machinery must be redesigned and new machinery built to make the laboratory tests of this type representative of service conditions. Mr. R. R. Moore's comment on the structural characteristics is very pertinent. Structural changes should always be studied in tests of this character. If the laboratory test fails to reveal structural conditions developed in service it should be carefully reconsidered.

I wish to thank all those who have commented upon this paper for their interest and the very valuable points that have been raised.

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[1] Lucas, *Transactions*, Am. Inst. Mining and Metallurgical Engrs., February, 1927.

[2] Schumacker and Nix, Pamphlet No. 1636-E, issued with *Mining and Metallurgy*, February, 1927.

[3] Schumacker and Bouton, *Journal*, Am. Chemical Soc., July, 1927.

PHYSICAL PROPERTIES AND METHODS OF TEST FOR SHEET BRASS

By H. N. Van Deusen,[1] L. I. Shaw,[2] and C. H. Davis[3]

Synopsis

This paper covers an investigation which was undertaken to secure a simple and reliable method of test of thin-sheet non-ferrous metals. Considerable attention was given to the Rockwell tester, which, as a result of this work, has been found satisfactory for use as a specification instrument for brasses 0.020 in. and thicker, when used under standardized methods of test and calibrated in terms of a standard instrument. Other tests, such as Meyer's analysis and the Amsler reverse bend test, show some promise of adaptation to this problem, but further development is necessary.

Physical data are given on complete series of samples of high brass sheet in a fairly wide range of thicknesses and tempers. Some data are also given on clock brass sheet. On the basis of these data and experience data obtained from commercial shipments of material, preliminary requirements were drawn up for these two grades of brass, subject to such changes as further experience may justify. Similar investigations are in progress on bronzes and nickel silvers.

Introduction

The investigation which this paper covers was undertaken because of the need for a simple method of test which would afford a more definite determination of the properties of thin-sheet non-ferrous metals than any which has yet been developed. Large quantities of brass, nickel silver and phosphor bronze are used in the manufacture of telephone apparatus as structural members, springs, and bearings. Because of space limitations, the parts are necessarily small; many are formed into irregular shapes; spring parts must maintain accurate adjustments and have long fatigue life; certain other parts must resist wear. All requirements are steadily becoming more exacting because of the increasing complexity of telephone systems and it, therefore, became necessary to insure more closely the uniformity of the material used in the apparatus.

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[1] General Development Laboratory, Bell Telephone Laboratories, Inc., New York City.

[2] Development Engineer, Western Electric Company, Inc., Chicago, Ill.

[3] The American Brass Co., Waterbury, Conn.

The methods of test developed to check the uniformity of materials are of equal concern to both consumer and supplier. Both are interested in a test rapid enough for use in inspection work and also so simple that specially trained men are not necessary for the actual testing. Due to the large amount of material that must be inspected it is necessary that any tests used require little time to apply and be adaptable to modern production methods. Furthermore, close agreement in test results is necessary. Since no test method was available fulfilling these requirements, it became necessary either to develop new methods of test or to modify existing methods. In addition, limits were desired that would represent the best quality of material obtainable consistent with commercial mill practices.

As a result of this need a cooperative program of tests was laid out which would lead to the drafting of requirements on thin-sheet metals. Those cooperating in this program were associated in the production and use of such material, and each member had already done preliminary work which evidenced his interest in the problem. The group was limited to only one producer and one consumer in order that the work might be expedited, and the results that have obtained in the relatively short time the investigation has been under way appear to justify the plan.

The results of the investigation, so far as it has progressed, are given herein for such value as they may have to the industry. It is hoped that this work may act as a stimulus to others interested in this problem so that ultimately definite standards may result, including the refinements demanded by modern industry.

SCOPE OF INVESTIGATION

In investigations of this character the tensile strength test is usually considered the basic mechanical test for cold-worked material. However, where large quantities of material are to be inspected, it is desirable to have a supplemental test which is simpler and more quickly performed. The scleroscope had been used for this purpose but had never proved entirely satisfactory, although at the time it was adopted, it was the only instrument available for rapid testing. Early work led to the consideration of the use of the Rockwell test as a possible solution of the problem. The reverse bend test, using the Amsler type machine and Meyer's analysis of the Brinell test, were also investigated. In addition, some Olsen and Erichsen ductility tests were made to supplement work which had been done by previous investigators.

In the case of the Rockwell tester, "round-robin" tests were made by the various laboratories cooperating in this work to establish a technique of test. These tests were made on three rolling series specially prepared for this purpose. Complete scleroscope data were obtained on all of these round-robin tests.

After the technique of test had been established for the Rockwell tester, rolling series of high sheet brass were made in many thicknesses and tempers on which complete chemical and physical data were obtained. Chemical and physical data were also obtained on a rolling series of clock brass.

Experience data were obtained with the Rockwell tester and scleroscope by both supplier and consumer on identical samples from many months' shipments of material.

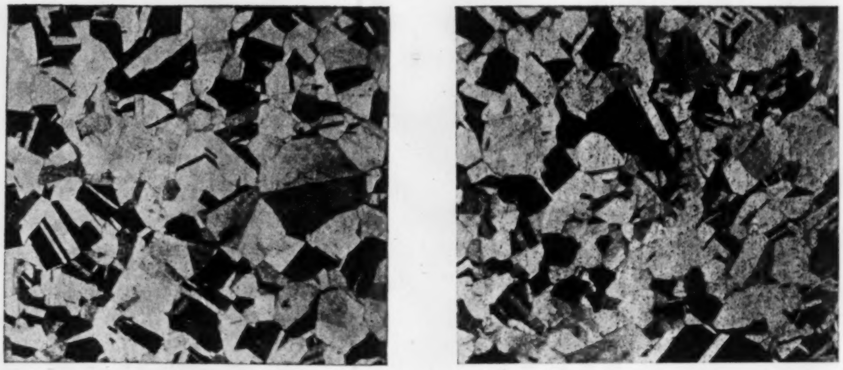
Based on these laboratory and experience data, commercial tensile and Rockwell limits for brass sheet were established on a trial basis.

Considerable work has also been done on nickel silver and phosphor bronze, but since this work has not been completed the data are not given except where they may serve to illustrate important characteristics of the methods of test.

METHOD OF HANDLING WORK

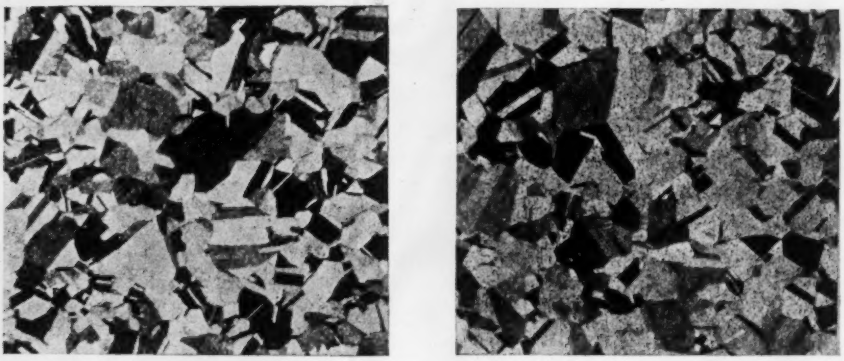
The round-robin tests on the Rockwell tester and scleroscope were made by the American Brass Co. (Waterbury, Buffalo and Kenosha Branches), Bell Telephone Laboratories, and two departments of the Western Electric Co. In addition, the U. S. Bureau of Standards and the Wilson-Maeulen Co. cooperated in testing one set of test blocks. All of the rolling series were furnished by the American Brass Co. from their Waterbury Branch. The physical tests on the high brass and clock brass rolling series were made by Bell Telephone Laboratories. The experience data on the several months' shipment of brass were obtained by the Kenosha Branch of the American Brass Co. and the Western Electric Co.

In addition to the authors, the following engineers took an important part in this investigation: Messrs. J. R. Townsend and W. S. Hayford of Bell Telephone Laboratories; W. H. Bassett, R. M. Tree and Alden Merrill of the American Brass Co.; W. A. Straw, H. F. Culver and H. R. Peery of the Western Electric Co. Messrs. N. E. Newton and W. H. Eastlake of The Northern Electric Co. of Montreal participated in all of the conferences that were held as the work progressed and gave the benefit of their experience.



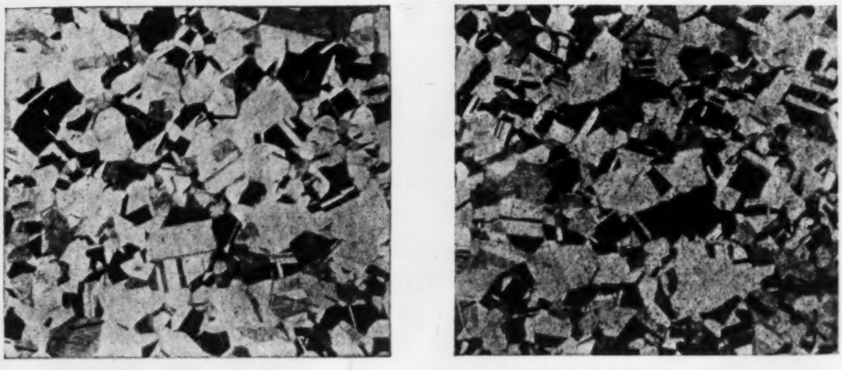
(*a*) 0.050-in. gage.

(*b*) 0.032-in. gage.



(*c*) 0.020-in. gage.

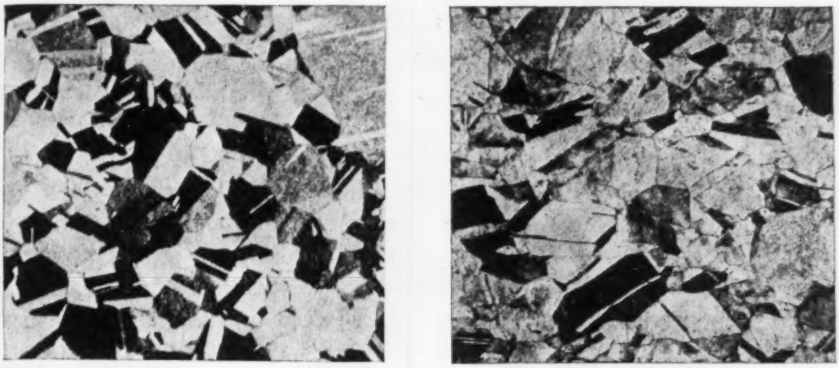
(*d*) 0.012-in. gage.



(*e*) 0.008-in. gage.

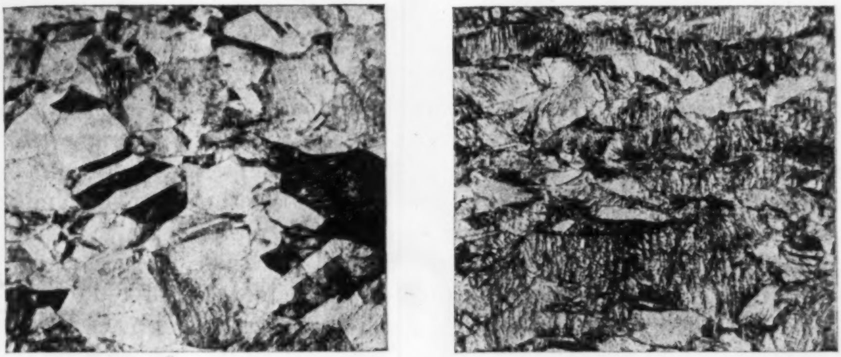
(*f*) 0.005-in. gage.

FIG. 1.—Grain Sizes of Annealed Bars in the High-Brass Rolling Series. Etched by Ammonia and Hydrogen Peroxide. (× 75.)



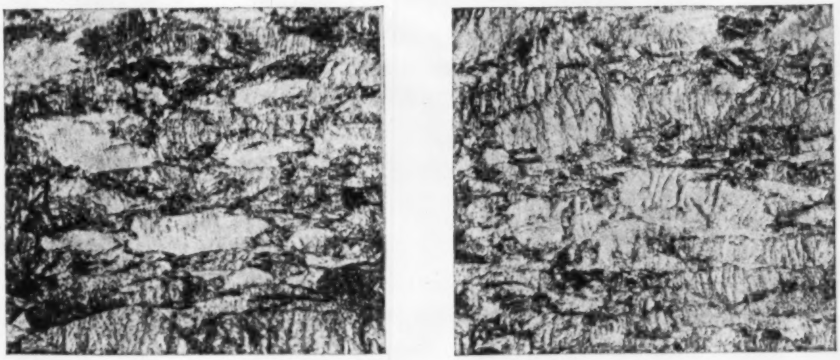
(*a*) Annealed.

(*b*) Reduced 21.0 per cent by cold rolling.



(*c*) Reduced 36.1 per cent by cold rolling.

(*d*) Reduced 50.2 per cent by cold rolling.



(*e*) Reduced 59.6 per cent by cold rolling.

(*f*) Reduced 67.5 per cent by cold rolling.

FIG. 2.—Structures of One High-Brass Series 0.064 in. Gage Reduced in Thickness by Cold Rolling from 0 to 67.5 per cent. Etched by Ammonia and Hydrogen Peroxide. (× 75.)

MATERIAL

Three separate rolling series were used for the round-robin hardness tests. The first of these consisted of a group of materials so arranged as to give a complete range of the hardnesses desired. The second was high brass with a nominal composition of 65 per cent copper and 35 per cent zinc. The third was nickel silver with a nominal composition of 64 per cent copper, 18 per cent zinc, and 18 per cent nickel. Each of these series included eleven tempers ranging from annealed material to the hardness resulting from 10 B. & S. gage numbers reduction. The samples were approximately 6 in. square and $\frac{1}{8}$ in. thick.

Complete series of samples comprising a fairly wide range of thicknesses were obtained on high brass sheet in order to thoroughly evaluate the methods of tests. These series were prepared in the following B. & S. gages: Nos. 14, 16, 20, 24, 28, 32 and 36. In each gage the samples had the following hardnesses: Annealed and 2, 4, 6, 8 and 10 B. & S. gage numbers reduction. These rolling series, therefore, included forty-two samples covering the usual commercial range of hardness and the range of thickness of greatest importance in telephone apparatus. These series were made under regular mill conditions with careful supervision to insure representative rolling practice and with complete records of the operations special attention being given to accurate records of the percentage of reduction. Care was taken to have the metal given approximately a 50-per-cent reduction before annealing to insure a uniform grain growth. The temperature of annealing was equivalent to about 600° C. for one-half hour, giving material of about average properties. Representative photomicrographs such as shown in Figs. 1 and 2 were taken so that the anneal and structure could be checked. These series were rolled from four bars and chemical analyses were made on each bar. The composition was as follows:

	BAR NO. 1	BAR NO. 2	BAR NO. 3	BAR NO. 4
Copper, per cent.........	65.19	64.94	65.10	65.13
Zinc, " "	34.77	35.02	34.85	34.82
Lead, " "	0.01	0.02	0.02	0.02
Iron, " "	0.03	0.02	0.03	0.03

The samples were flat strips 10 ft. long and 6 in. wide.

The rolling series on clock brass was made in the following gages and tempers: Nos. 12, 14, 16 and 18 B. & S. gages and 2, 4, 6 and 8 B. & S. gage numbers reduction. The composition was: Copper 61.63 per cent, zinc 36.75 per cent, lead 1.57 per cent, iron 0.05 per cent. This material is so nearly like the high sheet brass in its prop-

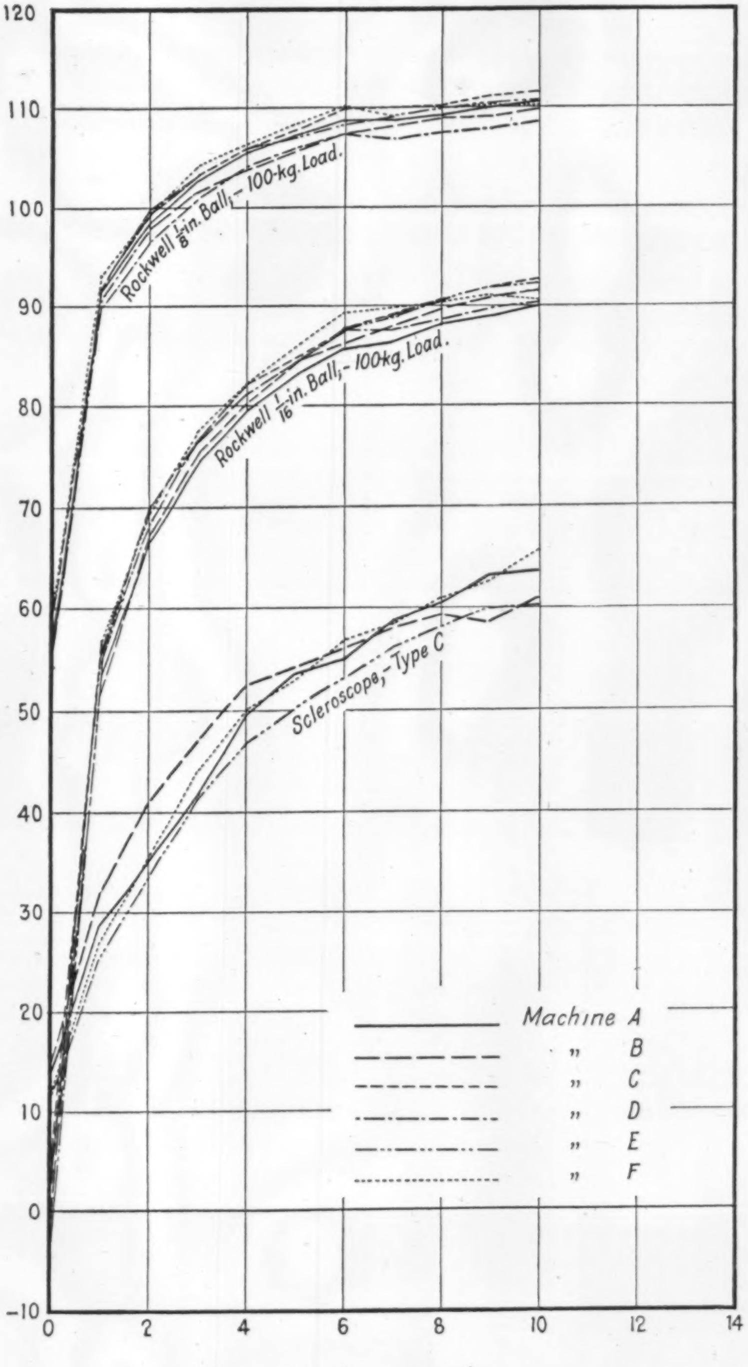


FIG. 3.—Brass "Round-Robin" Series. Comparison of Rockwell and Scleroscope Testers, Rockwell Manually Operated.

erties that the data collected when added to that already available were considered sufficient for the preparation of a set of requirements.

METHODS OF TEST

Scleroscope Hardness Tests:

Previous to this investigation Bell System specifications on nonferrous materials were written in terms of scleroscope hardness, but considerable trouble was encountered between the suppliers and users of metal due to difficulty in checking each others readings. Because results could not be duplicated on two instruments, it was necessary to allow rather wide limits in each temper of material. This resulted in considerable overlapping in scleroscope limits of the tempers of materials accepted under these specifications. Experience had shown it to be impossible to make correction curves for any two instruments which would hold for any reasonable length of time, and if any replacements such as new hammers were necessary the calibration was changed.

In order to obtain more definite information as to what could be expected from the scleroscope, comparisons were made between four type "C" sclerscopes located in four laboratories using the same samples of materials. Great care was taken in preparing the samples so that they were flat and free from any dirt and roughness. The instruments were in good commercial adjustment and were employed in the usual manner, the magnifier hammer being used. A single thickness of material was used in each case. The results of one of these comparison tests are shown in Fig. 3. Two other such comparisons were made incidental to the work being done to establish operating technique on the Rockwell tester, but the results shown here are typical. At least five readings were made on each sample and the average was used in preparing these curves. It will be noted that the readings on the different scleroscopes varied as much as eight points from each other on cold-rolled materials. The variations in readings between the machines follow no apparent law, one machine reading higher at one part of the scale and lower at another. It will also be noted from the curves that the range of the scleroscope is much smaller than that of the Rockwell on the same samples. In other words, the sensitivity of the scleroscope is considerably less than is that of the Rockwell.

An investigation was made of the type "D" recording scleroscope equipped with a universal hammer to obtain a comparison with the type "C" instruments. The conclusion reached was that there was little difference between the two instruments as regards precision.

It was not considered worth while, therefore, to make elaborate comparison tests. Hence, this type of instrument was not used in the round-robin tests.

In view of the difficulties encountered in using the scleroscope, which have also been brought out by previous investigators, no further effort was made to adapt it for use as a specification instrument.

Rockwell Hardness Tests:

While the Rockwell hardness tester has been used quite extensively and with considerable success in testing steels it has been used comparatively little for testing non-ferrous metals. Our experience previous to this investigation indicated that it might prove satisfactory as a specification instrument for non-ferrous metals. Considerable work had already been done by various laboratories to determine the limitations of this machine. Furthermore, the routine testing with the Rockwell of all incoming shipments of material had been instituted for the purpose of accumulating specification data. While each individual machine seemed to give results satisfactory to the user there was little information available as to what agreement could be obtained with machines in other laboratories. Figure 4 gives results of tests on identical samples with five different machines in the laboratories of one of the participating companies before any attempt was made to eliminate mechanical irregularities in the machines. While there was considerable difference between machines, there was a probability that by carefully going over the machines and establishing technique of test that these differences could be reduced.

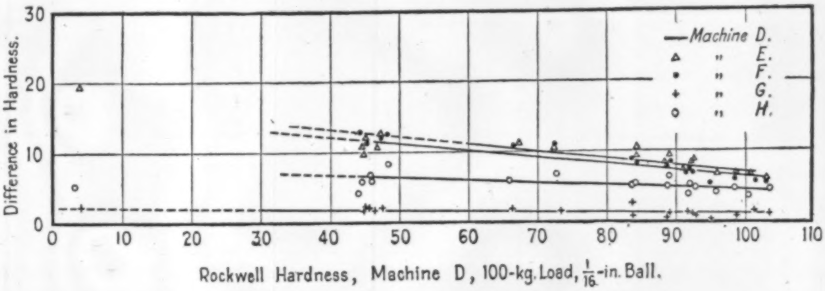


FIG. 4.—Preliminary Comparison of Rockwell Testers.

Before making any further comparative tests, careful study was made of each machine and various mechanical irregularities were eliminated. In order to get close comparative results the ball penetrators must be in good condition, the load must be applied without impact and at approximately the same rate in different machines, the value of the load must be the same, and the method of supporting the specimen across the anvil is important. Due to the lever system of applying load and measuring the amount of penetration of the material under this load, a slight amount of friction in the bearings may result in variations in readings which are not apparent without comparisons with standard blocks. A set of directions for calibrating the Rockwell tester was drawn up which called for a thorough check of these various features of the machine.

Other questions had to be settled, such as the size of ball penetrator to be used and the size of load. The possibility of varying the load and size of ball renders the Rockwell machine capable of broad application for testing purposes. Preliminary tests made to determine the most suitable combination of ball and load to be used indicated that the standard "B" scale of the instrument ($\frac{1}{16}$-in. ball and 100-kg. load) would be the most satisfactory for brass. However, it was decided to try also a $\frac{1}{8}$-in. ball with the same load. In our later work on nickel silver and phosphor bronze it was found that the 150-kg. load gave more satisfactory results with those materials. A larger ball is desirable to get readings on annealed brass and copper but with harder materials sensitivity of reading is increased by increasing the load and retaining the $\frac{1}{16}$-in. ball.

The question of the number of thicknesses of material to be tested was also given consideration. On very thin materials there is a distinct anvil effect which can be reduced or eliminated by superposing several samples on each other. However, this method of testing soon proved to be objectionable. The Rockwell tester does not distinguish between actual penetration of the material and descent of the penetrator from any other cause, and since one hardness number of the Rockwell machine corresponds to an 0.00008-in. movement of the penetrator, great care must be taken to insure that movement of the penetrator is due only to actual penetration of the material. Unless the spaces under the penetrator between two or more layers are completely closed by the 10-kg. minor load, low readings will be obtained. With spring materials, which are frequently curved due to coiling, this condition is often encountered. Furthermore, in very thin materials there is a side flow so that the upper sheets fail to take their share of the load and tend to curl up around the penetrator in such a manner as to prevent the proper measurement of depth. There is also a possibility of including small particles of dirt between the specimens which would result in lower hardness readings. These matters must be very closely watched. An indentation in the anvil just barely visible on a polished surface in strongly reflected light will cause erroneous readings. It is necessary, therefore, that the anvils be sufficiently hard for the purpose. During the past year, harder anvils have been furnished and greatly improved results have been obtained in the testing of thin materials.

A comparison of the methods used by the laboratories in making tests indicated that personal factors might account for some of the differences in readings. One of these personal factors which appeared to be of major importance was the amount of time allowed for the drift of the penetrator after the major load is completely applied. It was the custom of some of the participating laboratories to allow this drift to continue until it had practically ceased while others removed the major load and read the hardness as soon as possible after the major load had been completely applied. This drift is very noticeable in the softer tempers of non-ferrous metals and may amount to as much as 10 Rockwell numbers. In general, it is more noticeable

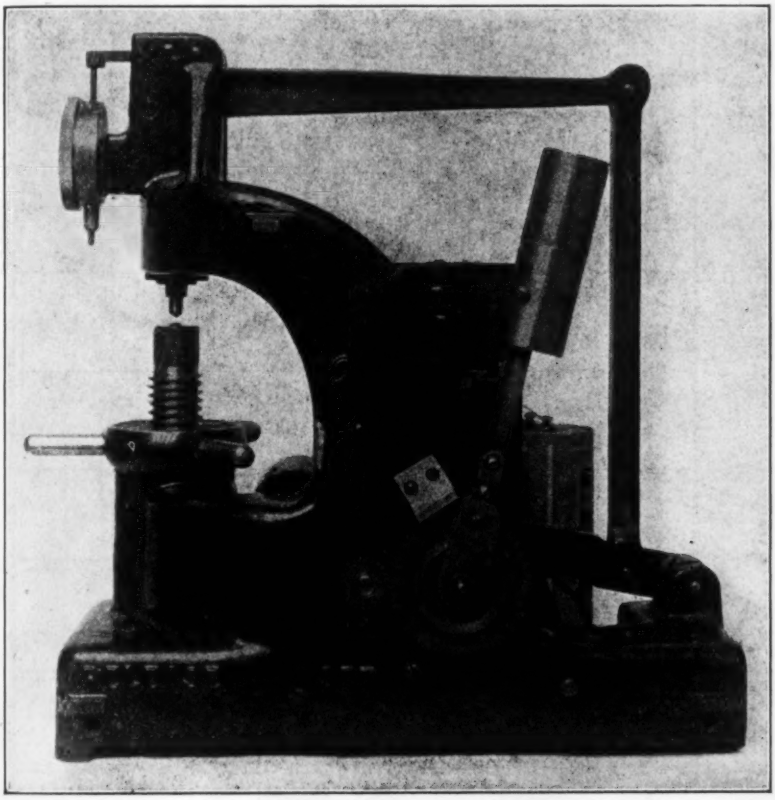


FIG. 5.—Rockwell Machine Showing Automatic Return Mechanism.

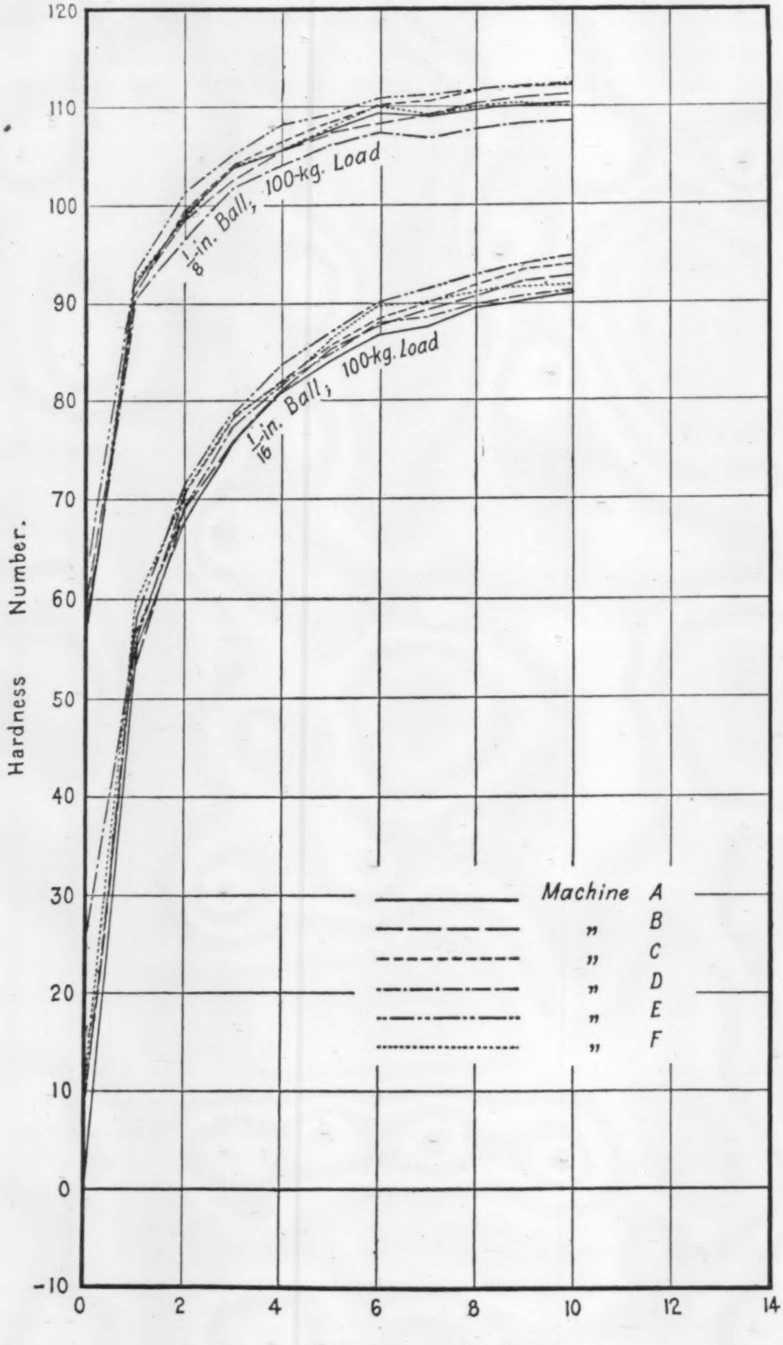


FIG. 6.—Brass "Round-Robin" Series. Comparison of Rockwell Testers Using Automatic Return Mechanism.

with non-ferrous metals than with steels. In order to eliminate this personal factor, an automatic return mechanism was developed and applied to the Rockwell tester which automatically removed the load about one second after it was fully applied. This device is illustrated in Fig. 5. While at first the use of this mechanism seemed necessary, further work showed that after a definite technique of test had been established, the question of drift was not an important one if directions were carefully followed, and the use of this device was discontinued. If such a device were built into the Rockwell tester it would eliminate this personal factor and make it possible for less experienced men to get more concordant results. While satisfactory results are now being obtained by "manual" operation, a wider use of the Rockwell machine for specification purposes may make it desirable to revert to the use of this automatic mechanism.

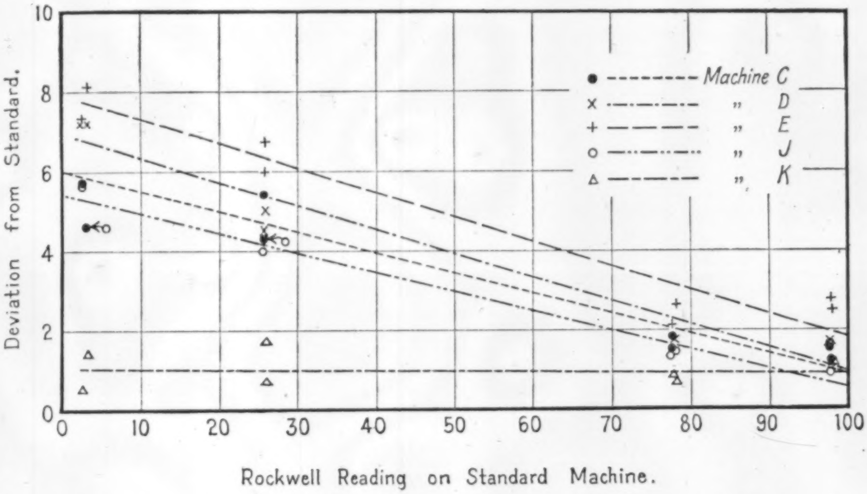


FIG. 7.—Comparison of Rockwell Machines on Standard Blocks.

As previously stated, three separate rolling series were used in the round-robin tests which were made after the various machines had been checked up. In each case tests were made with both $\frac{1}{16}$ and $\frac{1}{8}$-in. balls using the 100-kg. load with both the manual operation and the automatic return device. All tests were made on each of six machines located in the six different laboratories. Typical results of one of these series is shown in Fig. 3 which gives the results of a brass rolling series using both the $\frac{1}{8}$ and $\frac{1}{16}$-in. ball, the machine being operated manually. It will be noted that the greatest sensitivity is obtained by using the $\frac{1}{16}$-in. ball, and this was later adopted

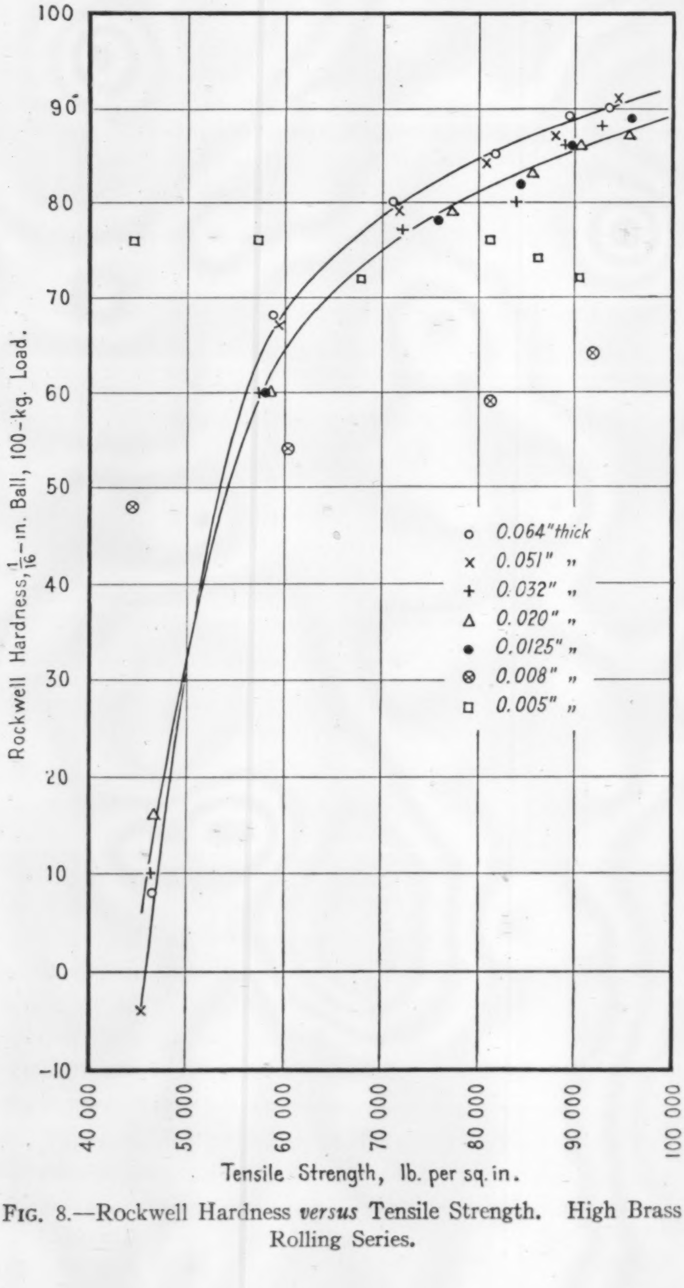


FIG. 8.—Rockwell Hardness *versus* Tensile Strength. High Brass Rolling Series.

as a standard ball for testing brass. Much better agreement between Rockwell testers than between scleroscope testers is also apparent.

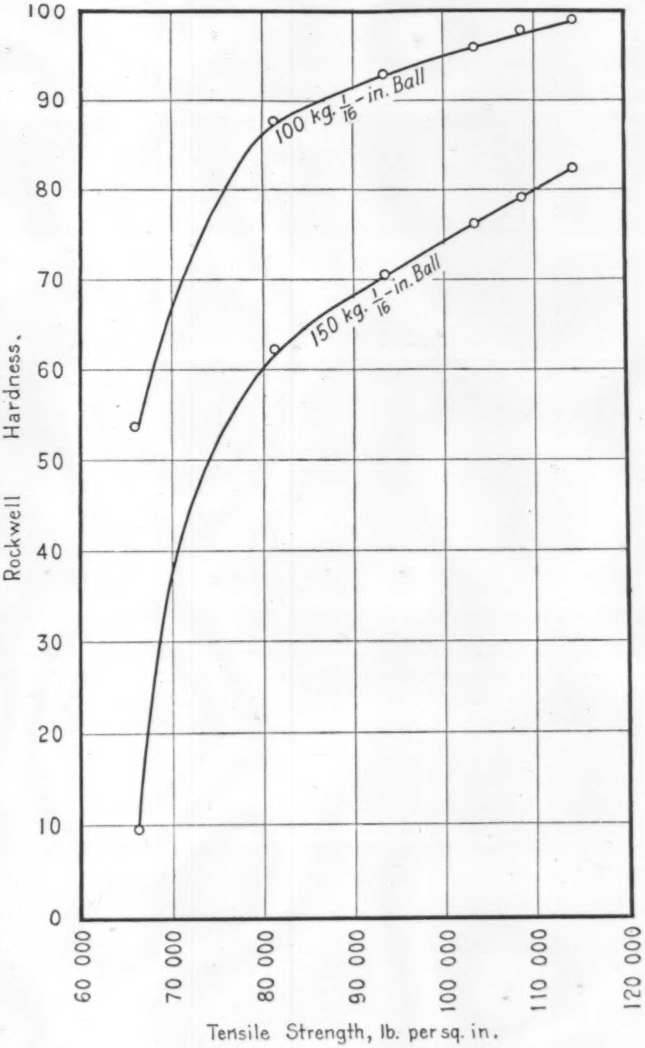


FIG. 9.—Rockwell Hardness. Nickel-Silver Rolling Series. Samples 0.064 in. Thick.

Results obtained on the same rolling series using the automatic return device are shown in Fig. 6. It will be noted that no advantage is to be gained from using this device if the manual tests are carefully made according to an established technique.

Even with the most careful checking of Rockwell machines it was found that the different machines varied in readings by as much as 8 numbers on the soft material and 2 numbers on the hard material. These differences are fairly constant, however, and each machine can be depended upon to retain its calibration for a considerable period. It is thus possible to provide a correction curve for each machine and give its readings in terms of the readings of a standard machine. It has been found convenient to have one machine reserved as a standard which will be used for the calibration of standard blocks. These blocks are non-ferrous and range in hardness from B-0 to B-100, it having been found necessary to check the entire range. Figure 7 shows the results of comparative tests on several of these standard blocks made on six Rockwell machines.

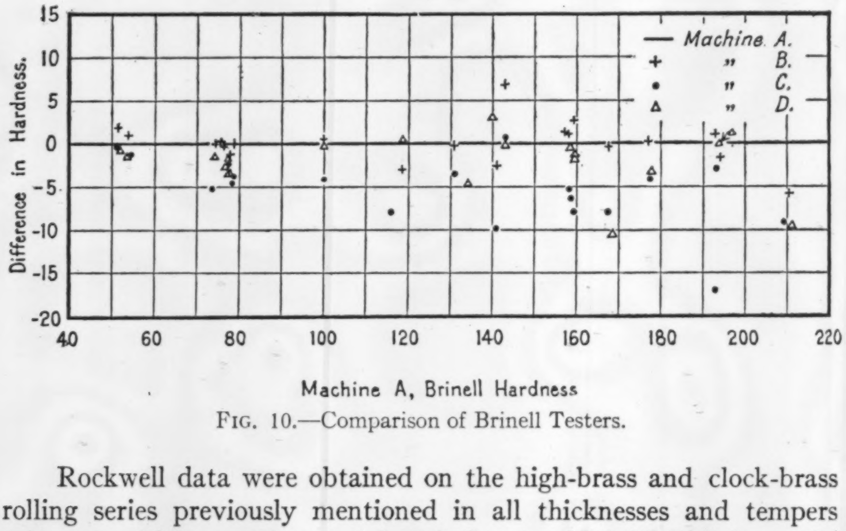


FIG. 10.—Comparison of Brinell Testers.

Rockwell data were obtained on the high-brass and clock-brass rolling series previously mentioned in all thicknesses and tempers using one thickness of material and the standard "B" scale. The plot of the results of these tests on high brass is shown in Fig. 8. These curves appear normal for thicknesses of 0.012 in. and above, but below this thickness the hardness readings bear no relation to tensile strength due to the anvil effect.

Phosphor-bronze and nickel-silver rolling series have been tested using the "B" scale. These tests show much smaller sensitivity for these materials than for brass. Sensitivity in the upper hardnesses for nickel silver may be increased by using a larger load with the $\frac{1}{16}$-in. ball. Figure 9 shows typical results obtained with the 100 and 150-kg. loads. It will be noted that the heavier load gives practically double the sensitivity of the lighter load. Further work is necessary to determine the best load for such materials. The use of a heavier load would, of course, increase the minimum thickness which could be tested successfully.

As a result of the experience gained in this investigation in using the Rockwell tester on non-ferrous materials a standard test procedure using this instrument has been drawn up and appears in Appendix I to this paper.

Meyer's Analysis of Brinell Test:

The Brinell hardness tester was not considered satisfactory for use in this investigation because it is not adaptable to thin materials. Furthermore, tests made by the different laboratories at the outset on standard Brinell hardness testers showed considerable variation between machines (see Fig. 10). It will be noted that there is no particular trend to the points on this curve, as there was with a similar test made on Rockwell machines (Fig. 4). While the Baby Brinell might be better than the standard Brinell for testing thin materials it is not in general use as a testing instrument and has not given rapid or consistent results.

Since the Rockwell test is not applicable to thicknesses below 0.012 in. on brasses and since there is a tendency for the Rockwell curves to flatten out in the harder tempers, particularly with the bronzes, indicating a decreased sensitivity, we were led to the study of Meyer's[1] analysis of the Brinell test, which has been reviewed recently by Hoyt[2] and others. For the purpose of completeness a brief review is given here. Meyer demonstrated that the Brinell number calculated for different loads and plotted load against Brinell number shows that the curve reaches a maximum and then falls off for all higher loads in a manner that is not at all consistent with the resistance of the metal to penetration. In order to give a more accurate measure of the hardness, Meyer suggested the use of the mean pressure that is supported by the metal, which was derived by dividing the load by the projected area of the ball indentation, namely:

$$P_m = \frac{P}{\frac{\pi d^2}{4}}$$

where P is the load and d the diameter of the impression. It was shown that the pressure plotted against the diameter varied in a regular manner with the load. The equation of the resulting curve is as follows:

$$P = a\,d^n$$

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[1] *Zeitschrift*, Verein Deutscher Ingenieure, Vol. LII (1908).

[2] *Proceedings*, Am. Soc. Steel Treating, Vol. VI (1924).

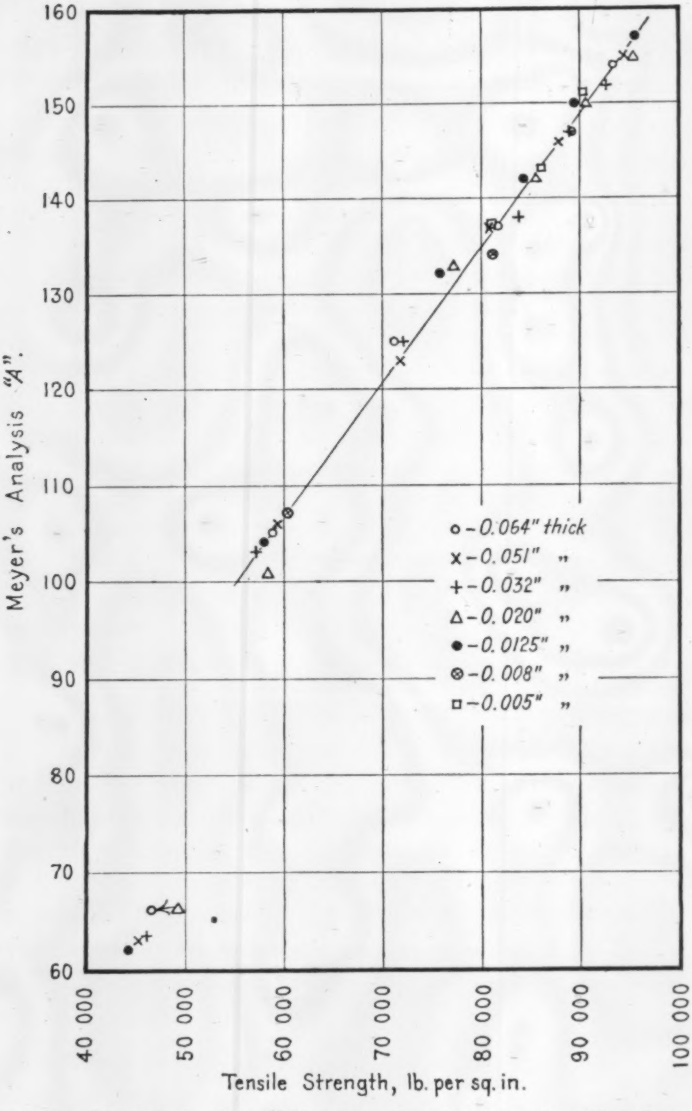

FIG. 11.—Meyer's Analysis "*A*" *versus* Tensile Strength. High-Brass Rolling Series.

If the load is plotted as abscissa against the diameter of the impression as ordinate on double-cycle log paper, a straight line results from which constants a and n may be obtained directly from the

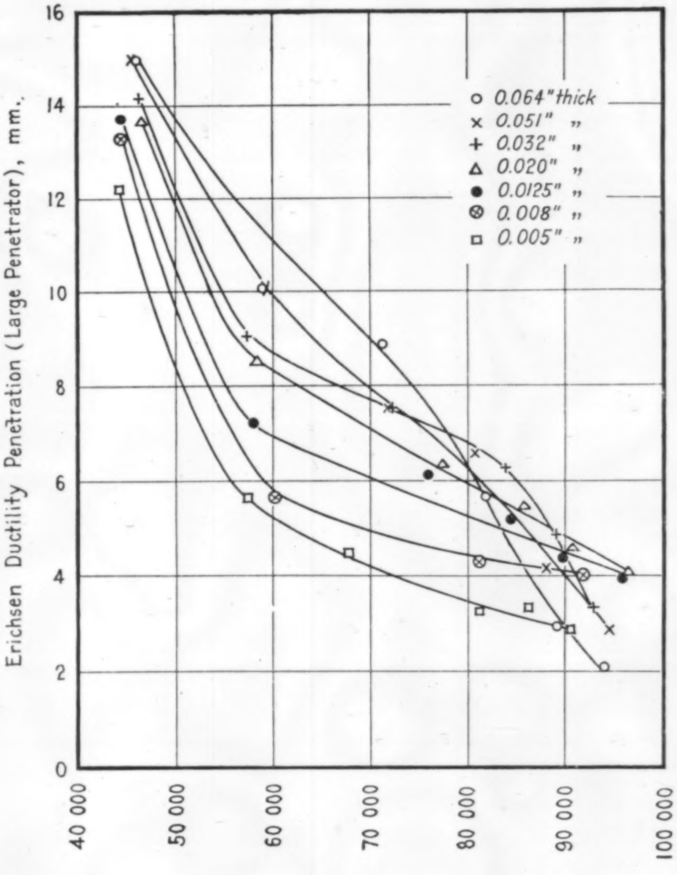

FIG. 12.—Erichsen Ductility *versus* Tensile Strength. Large Penetrator. High-Brass Rolling Series.

plot. Experience in measuring the hardness of a number of metals has shown that a is equivalent to the resistance of the metal to penetration.

We were interested to determine if Meyer's analysis could be used in a practical way and extended to sheet non-ferrous metals of all hardnesses and thicknesses. Since Meyer's analysis is based on fundamental considerations that do not involve the thickness of the metal under test there is no theoretical reason why this may not be done. In working out this problem it was found that a $\frac{1}{16}$-in. steel ball and loads up to 100 lb. were entirely satisfactory. The specimens of metal were placed between the compression jaws of a 100-lb. Schopper tension testing machine, the ball being mounted in the upper jaw. The diameters of the impressions were measured by a Société Genevoise Star Comparator which has a precision of one micron. Figure 11 shows the value of the constant a plotted against tensile strength for all hardnesses and thicknesses of the high-brass rolling series. A straight line relationship is found to exist between Meyer's constant a and the tensile strength, regardless of thickness.

Work done thus far on Meyer's analysis shows it to be applicable to accurate hardness measurements. However, this is distinctly a laboratory method and requires a careful workman to get satisfactory results. It may be possible to put this on a more practical basis and efforts are being made in that direction.

Ductility Tests:

In order to cover all available methods of testing, the high-brass rolling series was tested on the Erichsen and Olsen ductility testers. The material was cut in 3-in. squares and was tested on both these machines in the manner recommended by the makers in their instruction sheets. Both the large and small penetrators were used, but difficulty was experienced with some of the thicker samples due to the tendency for the material to shear. Typical results obtained by this method are plotted in Fig. 12. The thickness of the material being tested exerts considerable influence on the results and the curves for the thicker material cross those for the thinner. Similar results have been found in previous investigations[1] and show that the test is not suited to strict control of the material. It was also found in this investigation that different machines do not check one another.

The speed of testing, the lubrication of the metal and the amount of play allowed for the movement of the material over the face of the die exert a decided influence on the results obtained on these machines. For the reasons above noted, the ductility testers are not considered satisfactory for specification work.

Bend Test:

Some early work led to the consideration of the reverse bend test machine manufactured by the Amsler Co. (see Fig. 13) for distinguishing between materials which could or could not be successfully used in various manufacturing operations. This test consists in clamping a strip of material between blocks, the tops of which are rounded with radii in proportion to the thickness of the material being tested. The strip is put under tension and is then bent back and forth through 90 deg. over these radius blocks until fracture occurs.

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[1] C. H. Davis, "Testing of Sheet Brass," *Proceedings*, Am. Soc. Testing Mats., Vol. XVII, Part II, p. 164 (1917).

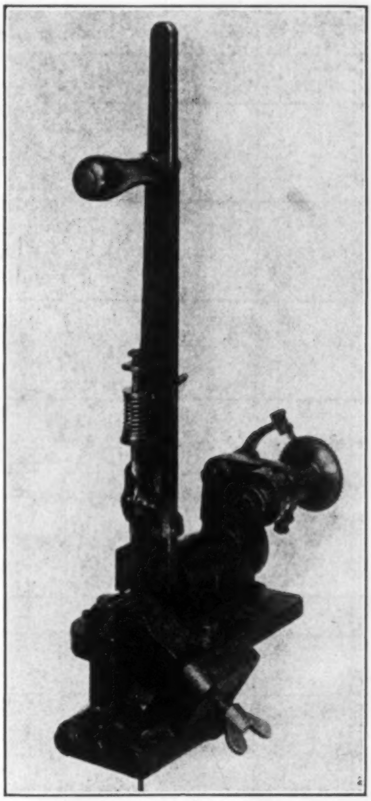

FIG. 13.—Amsler 90-deg. Reverse Bend Test Machine.

The machine as received had radius blocks, the radii of which were inaccurate. Accordingly, new blocks were made in which the radii were ground to an accuracy of 0.001 in. Further modifications were necessary in order to hold these blocks in proper alignment.

A study was made to see what ratio of block radius to thickness of material would give the best results, and it was found that this ratio should be somewhere near 5 to 1. If too large a ratio is used an excessive number of bends will be necessary to fracture the specimen, and if too small the test will lack sensitivity. It was found that if a strip of metal were tested over several different radii and the number of bends plotted against this ratio of radius to thickness

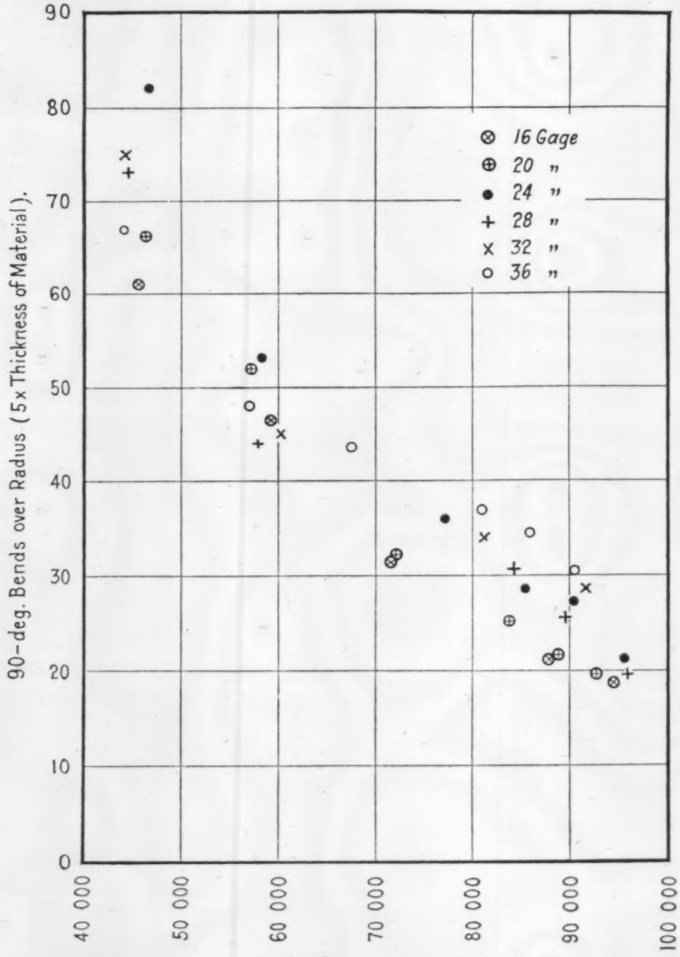

FIG. 14.—Bending *versus* Tensile Strength. High-Brass Rolling Series.

of material on log-log paper, the resulting line would be straight. It is possible, therefore, to use radius blocks of convenient sizes and interpolate the results to a chosen ratio. This reduces the number of blocks necessary for making complete tests on all thicknesses of materials.

The high-brass rolling series was tested on this machine using a ratio of radius to thickness of 5 to 1. The results are plotted in Fig. 14. This test seems to be considerably affected by the thickness of the material and rather wide variations are found between tests made on the same piece of material. The results obtained apparently depend on the qualities of the material above the elastic limit and close enough correlation between the tensile strength and the number of bends over all thicknesses and hardnesses of material has not been obtained. Further efforts are being made, however, to work out the difficulties with this type of test.

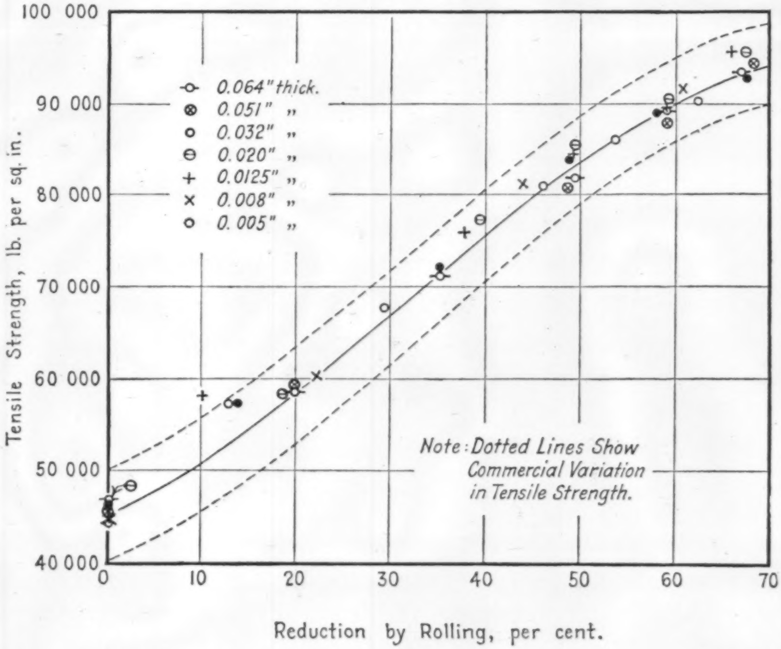

FIG. 15.—Tensile Strength *versus* Reduction by Rolling. High-Brass Rolling Series.

Tension Test:

As stated previously, the tension test is considered the basic mechanical test for cold worked materials. Complete data on tensile properties were therefore obtained on the high-brass and clock-brass rolling series. All tests were made in accordance with the standard testing procedure developed for use in the Bell System and given in Appendix II. Three tension test specimens were made from each sample. The tensile strength, proportional limit, percentage of elongation in 2 in. and modulus of elasticity were determined for each sample of the high-brass rolling series. The specimen 14 in. in length was used which allowed the use of an 8-in. gage length Anderson[1] extensometer. The shorter specimen with the 2-in. gage length was used for the clock brass and all later work. The tests were made on a tension testing machine of 10,000 lb. capacity where the maximum load required to break the specimen was more than 600 lb. and the remaining specimens were tested on a 600-lb. testing machine.

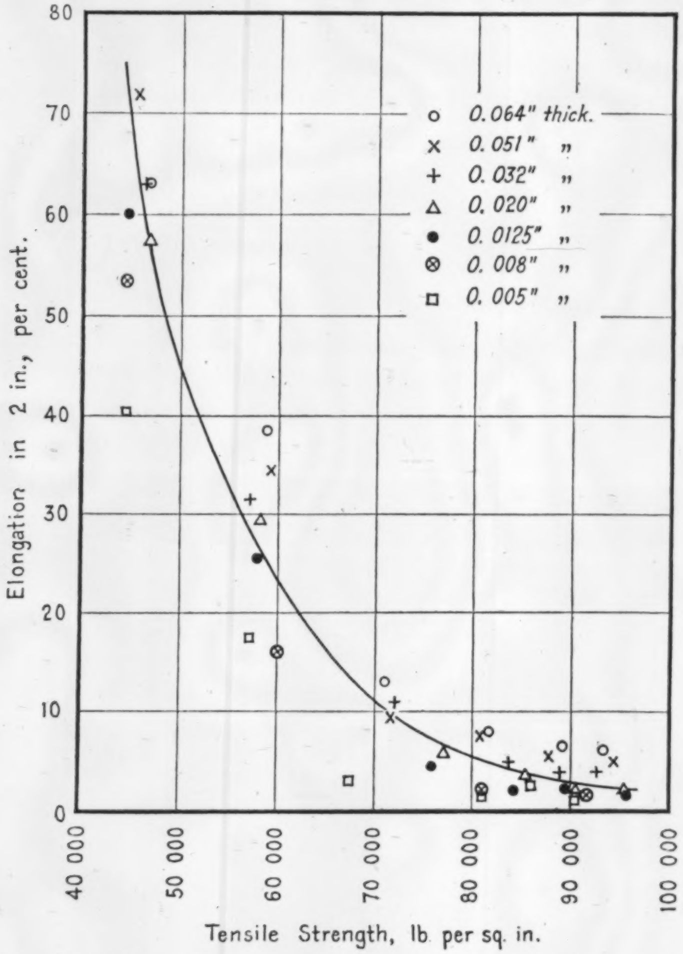

FIG. 16.—Percentage Elongation *versus* Tensile Strength. High-Brass Rolling Series.

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[1] H. A. Anderson, "Tension Tests of Thin Gage Metals and Light Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 990 (1924).

The machines were carefully calibrated, the load indications being correct to less than ½ lb.

The tensile strength and percentage of elongation on the high-brass sheet rolling series are plotted in Fig. 15 and 16, respectively.

TABLE I.—PHYSICAL TESTS ON HIGH-BRASS SHEET ROLLING SERIES.

B. & S. Gage	Thickness, in.	Numbers Hard	Percentage Reduction by Rolling	Proportional Limit, lb. per sq. in.	Tensile Strength, lbs per sq. in.	Elongation in 2 in. per cent	Scleroscope Hardness, Diamond Magnifier Hammer, One Thickness	Rockwell Hardness, "B" Scale, One Thickness	Meyer's Analysis "A," $\frac{1}{16}$-in. Ball. 1 mm. in diameter indentation, kg.	Erichsen Ductility, Large Penetrator, mm.	Erichsen Ductility, Small Penetrator, mm.	Olsen Ductility, Large Penetrator, in.	Olsen Ductility, Small Penetrator, in.
No. 36	0.0058	Annealed	0	9 500	44 200	40	15	76	80	12.20	3.17	0.462	0.113
No. 36	0.0055	2	12.7	33 000	57 200	17	33	76	102	5.64	2.38	0.202	0.064
No. 36	0.0058	4	29.25	32 000	67 600	3	41	72	110	4.48	2.17	0.149	0.062
No. 36	0.0054	6	46.0	38 000	81 000	1.5	50	76	137	3.27	1.93	0.128	0.062
No. 36	0.0057	8	53.6	39 000	86 000	2	54	74	143	3.35	1.88	0.122	0.048
No. 36	0.0060	10	62.5	35 500	90 400	1	58	72	151	2.86	1.86	0.107	0.046
No. 32	0.0087	Annealed	0	7 900	44 400	53.5	13	48	83	13.24	3.37	0.488	0.116
No. 32	0.0078	2	22.0	31 600	60 300	16	36	54	107	5.63	2.41	0.209	0.060
No. 32	0.0090	6	43.75	29 200	81 200	2	47	59	134	4.30	2.37	0.153	0.061
No. 32	0.0085	8	60.8	40 800	91 800	1.5	53	64	160	4.00	2.10	0.135	0.057
No. 28	0.0127	Annealed	0	8 300	44 500	60	9	13	62	13.69	3.73	0.542	0.129
No. 28	0.0144	2	10.0	38 800	58 000	25.5	24	60	104	7.21	2.94	0.262	0.087
No. 28	0.0135	4	37.8	30 500	75 900	5	31	78	132	6.11	2.76	0.209	0.074
No. 28	0.0127	6	49.2	40 300	84 400	2	38	82	142	5.16	2.52	0.188	0.059
No. 28	0.0134	8	59.1	34 300	89 600	2	49	86	150	4.37	2.27	0.144	0.054
No. 28	0.0136	10	66.0	44 300	95 900	1.5	51	89	157	3.94	2.32	0.149	0.050
No. 24	0.0221	Annealed	0	12 000	46 600	58	10	16	66	13.62	3.79	0.532	0.119
No. 24	0.0204	2	18.4	23 600	58 300	29	25	60	101	8.51	3.22	0.308	0.089
No. 24	0.0199	4	39.3	32 000	77 200	6	33	79	133	6.37	2.87	0.233	0.071
No. 24	0.0202	6	49.5	31 000	85 500	4	40	83	142	5.44	2.65	0.204	0.058
No. 24	0.0210	8	59.4	31 300	90 500	2	44	86	150	4.59	2.35	0.178	0.052
No. 24	0.0211	10	67.6	30 000	95 600	2	50	87	155	4.07	2.26	0.163	0.046
No. 20	0.0334	Annealed	0	6 100	46 300	63	9	10	63.5	14.17	4.10	0.559	0.124
No. 20	0.0345	2	13.75	23 200	57 300	32	26	60	103	9.05	3.46	0.348	0.107
No. 20	0.0336	4	35.1	23 500	72 100	11	34	77	125	7.52	3.05	0.272	0.076
No. 20	0.0333	6	48.9	27 100	83 800	5	48	80	138	6.27	2.48	0.231	0.065
No. 20	0.0340	8	58.0	33 300	89 000	4	51	86	147	4.83	1.84	0.175	0.063
Bo. 20	0.0331	10	67.6	33 800	92 700	3.5	56	88	152	3.32	1.90	0.130	0.061
No. 16	0.0529	Annealed	0	8 300	45 500	70	10	−4	63	15.00	....	....	....
No. 16	0.0524	2	19.6	22 900	59 300	35	36	67	106	10.08	....	....	....
No. 16	0.0525	4	35.2	23 100	71 700	9.5	42	79	123	7.50	....	....	....
No. 16	0.0523	6	48.7	27 600	80 700	7.5	46	84	137	6.53	....	....	....
No. 16	0.0521	8	59.3	30 500	87 900	5.5	51	87	146	4.16	....	....	....
No. 16	0.0510	10	68.5	29 600	94 500	5	59	91	155	2.86	....	....	....
No. 14	0.0661	Annealed	0	7 900	46 600	63	13	8	66	14.99	....	....	....
No. 14	0.0651	2	19.6	23 600	58 900	38	34	68	105	10.05	....	....	....
No. 14	0.0662	4	35.1	21 600	71 100	13	36	80	125	8.92	....	....	....
No. 14	0.0648	6	49.4	22 000	81 700	8	39	85	137	5.66	....	....	....
No. 14	0.0664	8	59.0	30 600	89 300	6.5	47	89	147	2.97	....	....	....
No. 14	0.0648	10	67.1	28 300	93 500	6	49	90	154	2.08	....	....	....

Nearly a straight line relation exists between the tensile strength and percentage reduction by rolling and the values are grouped closely about this line. In the percentage elongation curve, it will be noted that the effect of thickness of material is quite pronounced. The curve is plotted to show the average result, but separate curves could readily be drawn for each thickness. This test also has little sensitivity in the harder tempers where the curve becomes asymptotic. The proportional limit is difficult to measure with any degree of accuracy on account of the personal factor involved in interpreting curves. The results in this case showed no definite relation to any of the other values.

The complete results of the high-brass and clock-brass rolling series are given in Tables I and II.

EXPERIENCE DATA

During and subsequent to the laboratory work covered in this paper, a series of data was obtained on regular commercial shipments of non-ferrous sheet material. About 700 lots of brass, nickel-silver and phosphor-bronze sheet in various tempers and grades were tested by both the producer and consumer on the Rockwell and scleroscope. The sheet ranged in thickness from 0.010 to 0.500 in. A representative sample about 6 in. long and from 1 to 6 in. wide was taken from each lot. Tests were made on identical samples, five readings being made with each instrument and the average taken. All tests were made by the inspectors who were normally responsible for the control of the material, even though they were not in all cases familiar with the operation of the Rockwell machine. This was of special importance in determining the effect of the automatic return device discussed heretofore. The Rockwell machines were, of course, put in good mechanical condition and the methods of test outlined in Appendix I were followed. The results obtained constituted an independent verification of the conclusion that the Rockwell was

TABLE II.—PHYSICAL TESTS ON CLOCK-BRASS SHEET ROLLING SERIES.

Sample Number	Thickness, in.	Temper	B. & S. Gage Numbers Hard	Reduction by Rolling, per cent	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Scleroscope Steel Magnifier Hammer, One Thickness	Rockwell "B" Scale, One Thickness	Meyer's Analysis "A," kg.
502-2	0.081	....	2	20.4	59 250	32	35	68	106
502-4	0.064	....	4	36.6	72 750	9	48	80	125
502-6	0.050	....	6	49.8	83 700	6	54	85	137.5
502-8	0.039	....	8	60.4	90 000	4	58	88	152
340	0.143	Soft	..	....	45 900	62	15	21	67
341	0.081	Quarter Hard	..	....	51 400	40	25	56	86
414	0.040	Half Hard	..	....	61 800	25	35	70	106
412	0.079	"	..	....	64 500	23	40	77	119
342	0.033	"	..	....	70 200	14	39	78	125
190	0.020	"	..	....	64 600	13	..	65	113.5
415	0.040	Hard	..	....	74 300	8	46	82	127
413	0.078	"	..	....	74 800	8.5	45	84	125
191	0.032	"	..	....	74 500	5.5	..	79	123.5

NOTE.—502 samples were rolling series from American Brass Co.

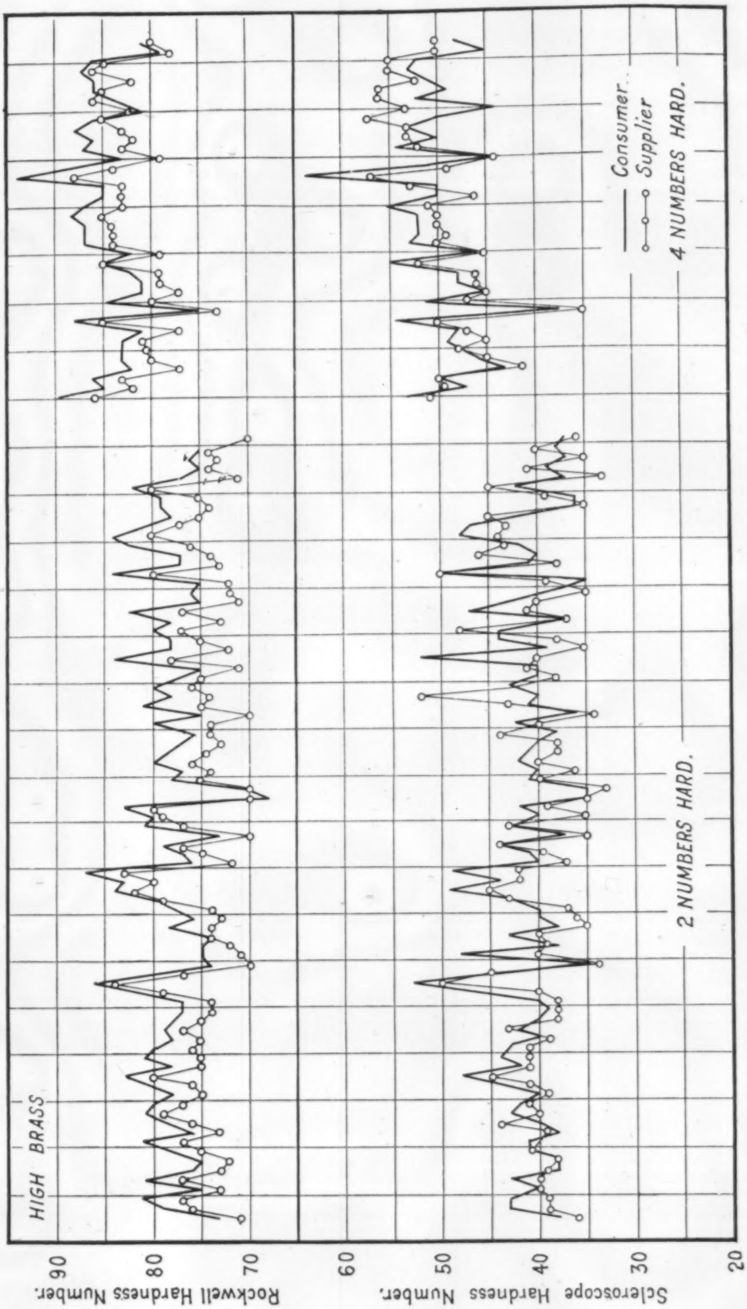

FIG. 17.—Typical Comparisons of Rockwell and Scleroscope Machines in Commercial Tests on Brass.

more sensitive but much less subject to variation and personal error than the scleroscope.

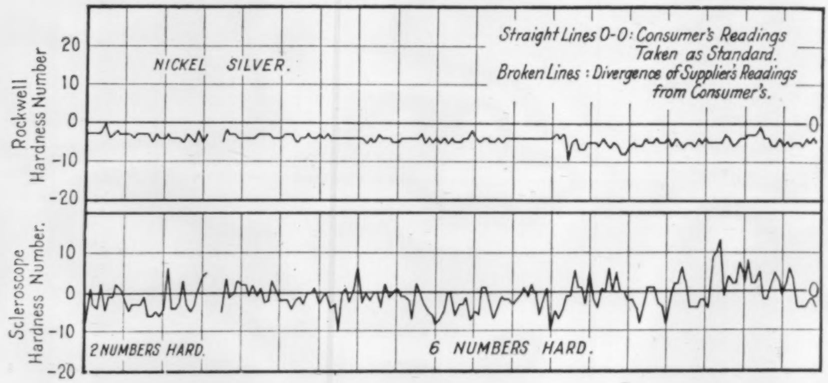

FIG. 18.—Typical Results Showing Differences in Rockwell and Scleroscope Machines in Commercial Tests on Nickel Silver.

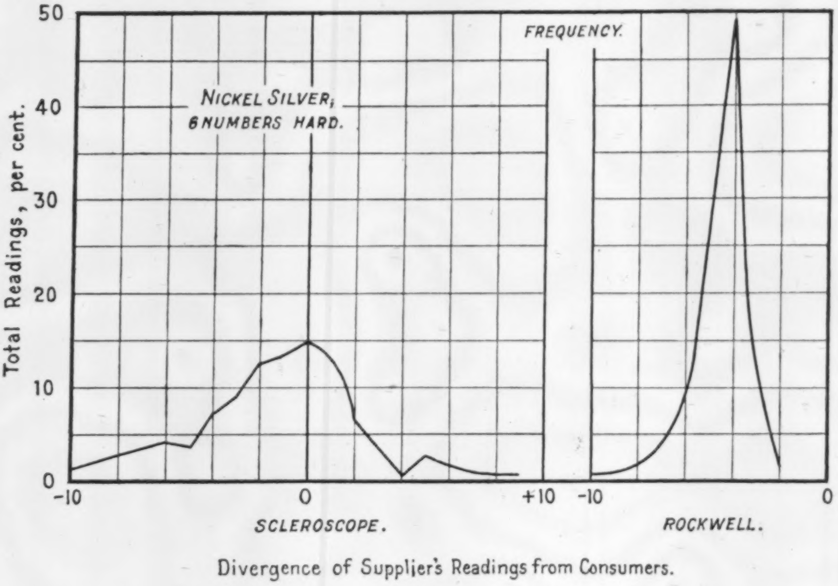

FIG. 19.—Typical Results Showing Frequency of Occurrence of Degrees of Difference Between Two Machines.

Figure 17 is representative of the results obtained, showing a direct comparison of the Rockwell and the scleroscope readings on two tempers of high brass. It illustrates the consistency of the readings of the two Rockwell instruments as compared with the two scleroscopes. Figure 18 represents similar results obtained on nickel silver plotted to show directly the differences between readings on the two machines of each type, one being assumed as a standard. Figure 19 shows the frequency of occurrence of the various degrees of difference between the two instruments, giving results for both the Rockwell and the scleroscope. One of the instruments was taken as a standard and the variations of the other instrument from it are plotted to left or right as they are negative or positive. The frequency of occurrence of differences of the magnitudes represented by the abscissas are plotted as ordinates in per cent of the total readings made on the group of samples for which the curve is drawn.

TABLE III.—PRELIMINARY REQUIREMENTS FOR HIGH BRASS SHEET

Thickness, in.	Temper	Numbers Hard	Reduction by Rolling, per cent	Tensile Strength, lb. per sq. in.		Rockwell Hardness "B" Scale (not to be used below 0.020 in.), One Thickness	
				Minimum	Maximum	Minimum	Maximum
0.040 and over	Soft	0	0	40 000[a]	50 000[a]	..	..
Under 0.040..........	Soft	0	0	40 000[a]	50 000[a]	..	..
0.040 and over	Quarter Hard	1	11.0	46 000	56 000	30	60
Under 0.040..........	Quarter Hard	1	11.0	46 000	56 000	30	56
0.040 and over	Half Hard	2	20.7	53 500	63 500	50	73
Under 0.040..........	Half Hard	2	20.7	53 500	63 500	47	69
0.040 and over	Hard	4	37.1	68 000	78 000	77	84
Under 0.040..........	Hard	4	37.1	68 000	78 000	73	80
0.040 and over	Extra Hard	6	50.0	79 000	88 500	84	88
Under 0.040..........	Extra Hard	6	50.0	79 000	88 500	80	85
0.040 and over	Spring	8	60.5	86 000	95 000	87	91
Under 0.040..........	Spring	8	60.5	86 000	95 000	84	88
0.040 and over	Extra Spring	10	68.7	89 500	98 500	87	92
Under 0.040..........	Extra Spring	10	68.7	89 500	98 500	85	89

[a] These figures do not represent the range of commercial annealing practice, but that of the final anneal before rolling.

In this typical case it is seen that about 50 per cent of the Rockwell readings from one machine are 4 points below those from the other machine. Application of a conversion factor would, therefore, move the curve to the right 4 points and very close checks between the two machines would result. The scleroscope curve shows that readings of the two instruments agree on only about 15 per cent of the total, and that in this case the differences are both positive and negative, thus not permitting the application of a conversion factor.

REQUIREMENTS

With the data obtained from the laboratory tests and with the experience data from actual shipments of material, together with information available from previous work, preliminary requirements were drawn up for the two grades of brass discussed herein. The tension test was used as the basis for determining the quality of the material. The two limiting curves shown on Fig. 15 were drawn in to represent the expected commercial variation in tensile strength and were taken from actual commercial experience covering a period of several years. These limits are commercial for material annealed between 450 and 750° C.

The Rockwell test is to be used as a ready method of test for rapid inspection purposes. The values for the Rockwell are intended to be those corresponding to the limiting values from the tension test.

TABLE IV.—PRELIMINARY REQUIREMENTS FOR CLOCK BRASS SHEET.

Thickness, in.	Temper	Numbers Hard	Reduction by Rolling, per cent	Tensile Strength, lb. per sq. in.		Rockwell Hardness "B" Scale (not to be used below 0.020 in.), One Thickness	
				Minimum	Maximum	Minimum	Maximum
0.040 and over	Soft	0	0	41 000[a]	51 000[a]	..	..
Under 0.040	Soft	0	0	41 000[a]	51 000[a]	..	..
0.040 and over	Quarter Hard	1	11.0	47 000	57 000	30	60
Under 0.040	Quarter Hard	1	11.0	47 000	57 000	30	56
0.040 and over	Half Hard	2	20.7	54 500	64 500	51	74
Under 0.040	Half Hard	2	20.7	54 500	64 500	48	71
0.040 and over	Hard	4	37.1	68 000	78 000	77	84
Under 0.040	Hard	4	37.1	68 000	78 000	73	80
0.040 and over	Extra Hard	6	50.0	79 000	88 500	84	88
Under 0.040	Extra Hard	6	50.0	79 000	88 500	80	85
0.040 and over	Spring	8	60.5	85 000	94 500	87	91
Under 0.040	Spring	8	60.5	85 000	94 500	84	88
0.040 and over	Extra Spring	10	68.7	88 000	97 500	87	92
Under 0.040	Extra Spring	10	68.7	88 000	97 500	85	89

[a] These figures do not represent the range of commercial annealing practice, but that of the final anneal before rolling.

It will be noted on Fig. 8 that the points for the Rockwell fall along two general lines depending on the thickness of the material. In this case, the break occurs between materials 0.032 and 0.051 in. in thickness. In our later work on the nickel silver and phosphor bronzes an intermediate gage was tested and it was shown quite conclusively that this break occurs at about 0.040 in. and was found in the case of all materials thus far tested. This characteristic of the Rockwell test is given consideration in the formulation of requirements for the different thicknesses of material. In formulating these requirements, it was not considered safe to use the Rockwell test on thicknesses below 0.020 in. since no round-robin tests had been made on thinner material to determine whether concordant results could be obtained.

However, our later experience indicates that satisfactory results may be obtained on brasses as thin as 0.012 in.

The requirements proposed for high brass and clock brass are given in Tables III and IV, respectively. They are presented here for discussion only. Further studies are to be made of grain size and the wear and fatigue characteristics and the results of such studies together with further commercial experience may make it desirable to change these proposed limits. While these requirements were drawn up primarily for thin materials, experience has shown that they will apply equally well to thicker materials. The Rockwell test is given as a convenient measure of the quality of material desired but the tension test is the standard on which the others are based and is to be used as a reference test. The numbers hard, temper and percentage reduction by rolling after annealing are given for information only. The composition of this brass is to be the same as that given in the Standard Specifications for High Sheet Brass (B 36 – 21) of the American Society for Testing Materials.[1]

CONCLUSIONS

The principal conclusions to be drawn from this investigation are:

1. The basic mechanical test for thin, cold-rolled non-ferrous sheet is the tension test, and it should be used as a reference test.

2. Under standardized method of test, when calibrated in terms of standard instrument, the Rockwell hardness tester is satisfactory for acceptance tests for cold-rolled brasses 0.020 in. and thicker. It also has a limited use for testing materials below 0.020 in. thick.

3. Meyer's analysis of the ball penetration test has been shown to be applicable to the laboratory testing of thin non-ferrous sheets, but further work is necessary to make it available for commercial testing.

4. The Amsler reverse bend test shows some promise of success, especially for metal under 0.020 in. thick, but the investigation is not complete.

5. The scleroscope and the Erichsen and Olsen ductility tests are not entirely suitable for specification work.

6. General requirements in terms of tensile strength and Rockwell hardness are given for cold-rolled high-brass sheet and clock-brass sheet. These are subject to revision after the further studies now in progress are completed.

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[1] 1927, Book of A.S.T.M. Standards, Part I.

APPENDIX I

STANDARD TEST PROCEDURE FOR ROCKWELL HARDNESS TESTS

Standard Machine

1. A Rockwell hardness tester will be reserved as the standard machine to calibrate standard test blocks. Comparison curves between the standard machine and any other Rockwell machine will permit the readings of such machine to be corrected to those of the standard machine. All specifications will be written in terms of the readings of the standard machine. Before any Rockwell tester is used in tests on metal to be supplied it shall be calibrated against this standard machine by means of the test blocks above mentioned and a calibration curve obtained.

Adjustments

2. (*a*) *Dash Pot.*—The dash pot on the Rockwell tester shall be so adjusted that the operating handle completes its travel in from five to ten seconds with no specimen on the machine and with the machine set up to apply a major load of 100 kg.

(*b*) *Index Lever Adjustment.*—As specified in the Rockwell tester instruction book, the following tests (and adjustments, if necessary) should be made.

"Put a piece of material on the anvil and turn the capstan elevating nut to bring the material up against the ball penetrator. Keep turning to elevate the material until the hand feels positive resistance to further turning, which will be felt after the 10-kg. minor load has been picked up and when the major load is encountered. When excessive power would have to be used to raise the work higher, take note of the position of the pointer on the dial. After setting the dial so that C-0 and B-30 are at the top then:

(*1*) If pointer stands between B-50 and B-70 no adjustment is needed.

(*2*) If pointer stands between B-45 and B-50 adjustment is advisable.

(*3*) If it stands anywhere else, adjustment is imperative.

"As the pointer revolves several times when the work is being elevated it is pointed out here that the readings mentioned apply to that revolution of the pointer which occurs as the reference mark on the gage stem disappears into the sleeve. The object of the adjustment is to see that the elevation of the specimen to pick up the minor load shall not be carried so far as to cause even a partial application of the major load, which to make a proper test, must be applied only through the release button."

"*To Make the Index Lever Adjustment, if Necessary.*—When the test piece is elevated until it starts to pick up the major load, loosen the lock nut of the screw through the index lever which carries at its lower end a small steel plate engaging the ball on the penetrator shaft while using a small screw-driver to firmly hold the screw until after the nut is loose. Then turn the screw very slightly and note result on position of gage pointer which should be at B-60 to B-70 before reclamping the screw with its lock nut."

(*c*) *Protection Against Vibration.*—If the bench or table on which the Rockwell tester is mounted is subject to vibration, such as felt in the vicinity of other machines, the tester should be mounted on a metal plate on sponge rubber at least 1 in. thick or on any type of mounting that will effectually eliminate vibration from the machine. Otherwise the pointer will penetrate farther into the material than when such vibrations are absent.

Cushioning of Latch.—If, when the operating handle is being returned to its normal position, the latch operates with such a snap as to noticeably change the position of the dial pointer, felt or rubber washers should be placed under the trip button in order to cushion this blow. If this snap is severe, difference in reading of several hardness numbers may result.

TESTING METHODS

3. The anvil used shall have a bearing surface for the material of about $\frac{3}{16}$ in. in diameter. It shall be hard enough so that no visible indentation is made when the thinnest material is tested.

Before using the machine it shall be operated several times on a piece of scrap material in order to firmly settle the penetrator, anvil and moving parts of the machine. This should be done every day before the machine is used.

Make a reading on at least one of the standard test blocks. This need not be done every day if it has been found that the machine does not change.

The Rockwell ball should be replaced by a new one occasionally and the operator should be on the lookout for any permanent deformation of the ball which will ordinarily be indicated by high readings on the standard test blocks.

When applying the minor load, the capstan screw should be turned up so that the pointer stops at "O" with a maximum variation of ± five divisions. The last movement of this screw must always be in such a direction as to elevate the specimen.

In applying the major load the operating handle shall be allowed to revolve without interference until the major load is completely applied. This may be observed in two ways, (1) when the pointer suddenly slows down, or (2) watching the weight arm to see that it is completely free from the control of the dash pot. When the major load has thus been completely applied, the operating handle shall be immediately brought back to the latched position without waiting for it to complete its revolution. This should be accomplished in less than two seconds after the major load is completely applied.

The test specimen shall be well supported to prevent errors due to the overhang of the specimen.

All tests shall be made upon one thickness of the material.

APPENDIX II

STANDARD TEST PROCEDURE FOR TENSION TESTING OF THIN SHEET METALS[1]

GENERAL

1. (*a*) These specifications cover general methods for testing the tensile strength of thin sheet, strip and flat wire materials for use in the manufacture of telephone apparatus. They shall form part of individual specifications covering sheet metals for specific purposes whenever so provided in the individual specifications.

NOTE.—By thin sheet material is meant material 0.05 in. or less, in thickness. This thickness lies in the range where it becomes necessary to abandon the use of the ordinary micrometer and to use the barrel type micrometer or a still more sensitive device in order to keep the probable error of measurement as low as possible.

(*b*) In case of any difference in the provisions of this and the individual specification, the provisions of the individual specification shall govern.

SPECIMENS FOR TENSION TESTS

2. *Selection of Specimen.*—The test specimens shall be chosen in such numbers and from such locations in each lot of material as to be representative of the quality of this material. The method of selecting specimens will be specified in the individual specifications when found necessary.

3. *Dimensions of Specimens for Tension Test.*—

(*a*) *Materials ½ in. or Less in Width.*—For strip or flat materials less than ½ in. wide, the specimen shall consist of the appropriate length cut from the material as furnished. For gage lengths of 2 and 8 in., the length of the test specimen shall be not less than 8 and 14 in., respectively.

(*b*) *Materials More Than ½ in. in Width.*—For materials over ½ in. wide, two alternative specimens are allowed.

(*1*) *Standard Specimen:*

The specimen which shall be regarded as standard shall have dimensions and be machined as shown in Fig. 20.

(*2*) *Alternative Specimen:*

This specimen shall be in the form of a strip up to 1½ in. in width, having parallel sides and having about 0.001 to 0.002 in. of the material removed from the edges of the specimen near the center by grinding with a fine-grained stone. This specimen shall have the lengths given above for materials less than ½ in. wide. It may only be used so long as good breaks (which occur at the point of reduced section and without tearing) are obtained and where the strength of the material does not approximate the acceptance limits. The milled specimens (see Standard Specimen above) are to be regarded as standard and are to be used where good breaks are not obtained with these wider specimens and as referee test specimens where the strength of the material approximates the acceptance limits.

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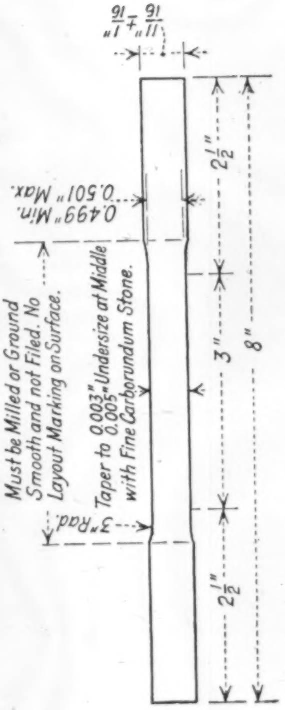
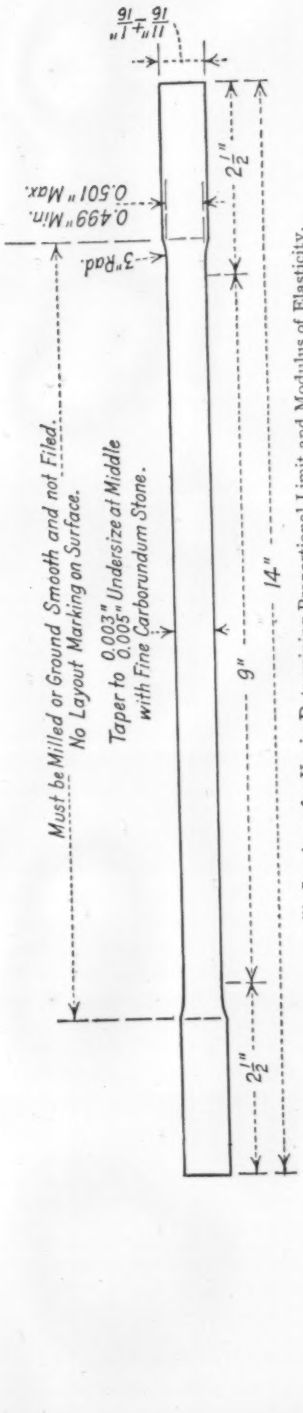
[1] As prepared and used by Bell Telephone Laboratories and Western Electric Co.


11"/16 + 1"/16
0.499" Min.
0.501" Max.
Must be Milled or Ground Smooth and not Filed. No Layout Marking on Surface.
Taper to 0.003"/0.005" Undersize at Middle with Fine Carborundum Stone.
3" Rad.
2½"
3"
2½"
8"

(a) Specimen for Use in Determining Tensile Strength and Elongation in 2 in.


11"/16 + 1"/16
0.499" Min.
0.501" Max.
3" Rad.
Must be Milled or Ground Smooth and not Filed. No Layout Marking on Surface.
Taper to 0.003"/0.005" Undersize at Middle with Fine Carborundum Stone.
2½"
9"
2½"
14"

(b) Specimen for Use in Determining Proportional Limit and Modulus of Elasticity.

FIG. 20.—Tension Test Specimens for Sheet Material Over ½ in. in Width.

4. *Cleaning of Specimens.*—Before being measured, each test specimen shall be thoroughly cleaned with carbon tetrachloride applied by rubbing with a soft cloth. When being measured, each specimen shall be wiped clear of dust immediately before being inserted in the measuring instrument.

5. *Measurement of Specimens.*—The dimensions of the specimen shall be measured between flat parallel surfaces of hardened steel, or other appropriate material, approximately $\frac{3}{16}$ in. in diameter which shall be applied in such a manner as to exert on the specimen, while the measurement is being taken, a normal pressure of approximately 16 oz.

6. *Calibration of Measuring Instrument.*—The measuring instrument shall be suitably adjusted and calibrated to indicate the distance between the measuring surfaces of the mandrels when exerting a normal pressure of approximately 16 oz. or 35 lb. per sq. in. of mandrel end area with an error not greater than ± 0.00005 in., *i. e.*, to the nearest 0.0001 in.

NOTES

1. When measuring very thin materials, the micrometer or measuring instrument should be adjusted and calibrated with the greatest practicable accuracy.

2. The measuring instrument should be calibrated preferably by means of a hardened steel gage block having flat parallel surfaces and of a thickness approximately equal to that of the material to be measured.

3. In order to obtain an accurate check, it is essential that the mandrel surfaces and faces of the gage be freed from dust immediately before being brought together.

4. If a barrel type micrometer indicating 0.0001 in. per scale division is used, care should be taken to have the instrument in good operating condition. The instrument can then be adjusted to give the first click of the ratchet at a mandrel pressure of 16 oz. ± 4 oz. by suitable adjustment of the ratchet spring, measuring the mandrel pressure by means of a small spring balance, having flat parallel contact surfaces, inserted between the mandrels. The micrometer screw and nut shall be so adjusted that no end play or shake of the spindle may be felt when the spindle is gently pushed back and forth in the direction of the axis of the screw.

5. In calibrating the micrometer against a standard gage block, or in measuring a specimen, the spindle shall be rotated slowly into contact with the work by means of the ratchet until the ratchet clicks once, be retracted about 0.002 in. and again slowly rotated into contact until the ratchet clicks once before reading is taken. In order to avoid exceeding the normal pressure of the mandrels in operating the micrometer, the mandrel shall be brought against the work at a rate not exceeding 0.001 in. per second at both first and second contacts given above. This rate is approximately the maximum rate at which the operator is able to count the half thousandths marks on the barrel as they pass the zero line on the frame.

7. *Method of Computing Cross-Section.*—

(*a*) *Material* $\frac{1}{2}$ *in. or Less in Width.*—For strip material of flat wire $\frac{1}{2}$ in. or less in width, the width and thickness shall be measured along the major axis of the specimen at the middle point and at distances of approximately 2 and 4 in., respectively, at each side of the middle point. The cross-section of strip and flat wire material shall be assumed to be a rectangle. The area of cross-section to be used in computing the tensile strength of the material shall be the product of the average of the five thickness readings and the average of the five width readings.

(*b*) *Cross-Section of Standard Test Specimen for Material Over ½ in. in Width.*—For the standard test specimen described in Section 3 (*b*), the width shall be measured at the point of minimum width as determined by trial measurements. The thickness shall be measured in a plane normal to the major axis of the specimen at the point of minimum width, at the middle and at each edge of the specimen. When measuring the thickness at the edge of the specimen, the outer edges of the contact surfaces of the measuring instrument shall be approximately $\frac{1}{32}$ in. inside of the edge of the material. The area of cross-section shall be taken as the product of the minimum width and the average of the three thickness readings above specified.

NOTE.—In some cases, it may be found that the thickness of the material near the fillets is so much less than the thickness in the plane of minimum width that the minimum area of cross-section occurs near the fillet. In measuring specimens, the operator should be on the lookout for this condition and, in case it is found, the width of the specimen at the point of minimum width should be further reduced by grinding with a stone until the minimum cross-section is brought to the middle of the specimen.

(*c*) *Cross-Section of Test Specimen Over ½ in. in Width.*—For specimens over ½ in. wide as described in Section 3 (*b*), the measurements shall be made as on the standard specimen, as described in Paragraph (*b*) above.

METHOD OF TESTING

8. *Testing Machine.*—

(*a*) *Design.*—Testing machines shall be of a design in which the applied load is balanced by increasing the lever arm of a movable weight or by some other means employing weights without the use of springs.

(*b*) *Method of Mounting.*—The testing machine shall be installed on a solid foundation of such a nature that the machine is maintained in a level position.

(*c*) *Range.*—It is desirable that the testing machine should be capable of applying and measuring a load about 50 per cent greater than the normal anticipated breaking strength of the test specimen.

(*d*) *Sensitivity.*—All bearings and knife edges of the testing machine shall be so proportioned and adjusted that the position of the beam, when balanced at the normal breaking load of the specimen, shall be perceptibly changed by a change of load of 0.1 per cent.

(*e*) *Accuracy.*—The machine shall be calibrated with dead weights so that a load equal to the breaking load of the material tested shall be indicated with an error of less than ±0.1 per cent.

(*f*) *Method of Applying Load.*—The mechanism for applying load to the test specimen shall be such as to advance the pulling head of the machine at a uniform rate, such as may be obtained by a motor drive operating from a well regulated source of power. The machine shall be capable of being operated at different uniform rates of speed, including the rate of approximately 0.05 in. per minute of the moving head. Unless otherwise specified in individual specifications, the maximum speed of the moving head in making a test shall be 0.025 in. per inch free length of specimen per minute.

(*g*) *Jaws.*—The jaws or grips of the testing machine shall be designed so as to produce and maintain axial alignment of the specimen without producing tearing stresses. The edges of the jaws shall be rounded so as not to noticeably deform the specimen near the borders of the region where the specimen comes in contact with the jaws.

DISCUSSION

Mr. Reinhardt Mr. J. A. Reinhardt.[1]—I should like to ask whether the consumers and manufacturers who took part in the investigation have adopted these standards in their specifications.

Mr. Shaw. Mr. L. I. Shaw.[2]—While we have been accumulating data for a period of a year and a half, we have not as yet incorporated the limits in our specifications. We know how the supplier is meeting the requirements and how we are checking with each other. I believe there is no reason for their not being adopted except that we have desired to accumulate data to see if the limits were satisfactory.

Mr. Clamer. The Chairman (*Past-President G. H. Clamer*[3]).—Has this matter been put up to the suppliers?

Mr. Shaw. Mr. Shaw.—No, it has not been submitted to them formally, but we have had informal talks with different suppliers. I think Mr. Bassett can say, as one supplier, how he feels about the acceptance of these limits.

Mr. Bassett. Mr. W. H. Bassett.[4]—This work is expected to form the basis of specifications. The existing specifications, which are based on the use of the scleroscope, have from the suppliers' standpoint, and I think probably also from the users' standpoint, proved a complete failure, and it was concluded that something should be done to form the basis for specifications for which we had more exact measurements than existed; consequently, the testing of these various machines which might be used as a standard measure of hardness. Of course you recognize that a large amount of work has been done beyond what has been reported. There have been informal discussions, as Mr. Shaw said, between the Western Electric Co. and the American Brass Co. and various other suppliers of material. I think that while the work has been very largely carried out by the Bell Laboratories, the Western Electric Co. and the American Brass Co., the other large suppliers of material are cognizant of what has been done and have expressed themselves as in sympathy with it. The hope is that this work will form the basis of a general specification for sheet brass material. I think possibly by another year Committee B-2 on Non-

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[1] Metallurgist and Assistant Chief Engineer, Western Clock Co., La Salle, Ill.

[2] Development Engineer, Western Electric Co., Inc., Chicago, Ill.

[3] President and General Manager, The Ajax Metal Co., Philadelphia, Pa.

[4] Technical Superintendent and Metallurgist, The American Brass Co., Waterbury, Conn.

Ferrous Metals and Alloys can borrow some of these data to place before the Society in further specifications. In the meantime, we assume that some of the producers and consumers will use them in specifications. Of course they are necessarily more or less tentative, but certainly form a better basis than anything we have had heretofore. **Mr. Bassett.**

MR. J. L. CHRISTIE.[1]—I should like to add to what Mr. Bassett has said. For the past two years or so the Bridgeport Brass Co. has used the Rockwell test as a laboratory check on mill procedure, not only on the hard-rolled brasses as spoken of here, but also on the annealed brasses; and while we have done very little in making brass to Rockwell specifications, we have found the instrument very reliable as a laboratory test controlling mill operations. **Mr. Christie.**

MR. SHAW.—In this paper are given very tentative limits for the Rockwell hardness. In a reasonable length of time we shall have purchase specifications using these limits as a basis, and in the same way anyone can take these data and write his own purchase specifications if he so desires. From our standpoint we would welcome anyone trying out these limits and seeing how they work. There may be some question as to the limits; it may be that they will have to be revised; they are only put out as something to try, and of course, the use to which the material is put may necessitate a change in the limits. We shall be glad to cooperate with either consumers or producers in any way that we can in this matter. **Mr. Shaw.**

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[1] Metallurgist, Bridgeport Brass Co., Bridgeport, Conn.

WEAR TESTING OF METALS[1]

BY H. J. FRENCH[2]

SYNOPSIS

Factors affecting the wear of metals are considered and some of the difficulties encountered in making both laboratory and service wear tests are discussed. Two testing machines used in the study of the wearing properties of metals employed for widely different purposes are briefly described and attention is given in particular to the initial surface condition and "filming" of metals in relation to the procedure employed in making wear tests.

The report is a brief summary of the present status of wear testing of metals and includes results of experimental work to show that reproducible results and information consistent with practical experience can be secured in the laboratory.

INTRODUCTION

Wear is defined in Webster's dictionary as the destructive effect of work or usage. This connotes what in every-day language is called "wear and tear," and does not take into account the nature, cause or degree of unserviceability produced. It is a very broad definition and may be conceived to include failure by fatigue, deterioration through corrosion or through instability at high temperatures, etc. As applied to metals in this discussion, the word wear is used in a narrower sense and considered to consist of the removal of metal at the surface of a solid body by friction with another body or bodies.

Probably more metal articles are ruined or made unserviceable by wear than by any agent other than corrosion. This is due to the ever-present tendency for wear to occur when two or more solid bodies in contact move with reference to each other. Hence, the problem of defeating wear is widespread and will be encountered in different forms and under widely varying conditions. While this discussion will be restricted to the wear produced by friction between solid bodies it should be noted that erosion by liquids or gases comes within the adopted definition.

Such cases as the wear of rails, tools, bearings and other machinery parts in which the friction is produced between two metallic surfaces, are well known. Another well-recognized type is that to which grinding, crushing and excavating machinery is subjected, in which the friction is produced between a metallic body or bodies and non-metallic abrasives. An interesting but unusual case which illustrates the broad aspect of the wear problem is shown in Fig. 1. The wire parts illustrated are "travelers" and "flyers" used in silk spinning. According to one manufacturer continuous passing of silk threads over the long horizontal section of the traveler or through the "eyes" of the flyers wears in two the wire from which these parts are made or cuts grooves in it which will be rough enough to injure the silk.

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[1] Published by permission of the Director, U. S. Bureau of Standards, Washington, D. C.

[2] Senior Metallurgist, U. S. Bureau of Standards, Washington, D. C.

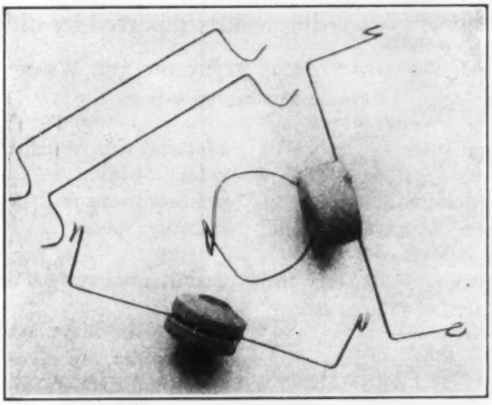

FIG. 1.—"Travelers" and "Flyers" Used in Silk Spinning.

Shown to illustrate the diverse nature of the wear problem for metals. The continuous passing of silk over the wires cuts grooves or wears them in two.

With the problem of the wear of metals so widely distributed and of such general interest it is, perhaps, difficult to understand why more attention has not been paid to wear testing. Probably one of the principal reasons is in the complexity of the problem and the expense and patience required to secure information of value. When an elaborate series of tests has been completed it is, often, even then not possible to say that one material is more resistant to wear than another, but only justifiable to point out that under certain restricted conditions one has been shown to be superior in certain respects to another.

The object of this paper is to point out certain important features that have been observed in experimenting with a number of wear testing machines. Particular attention will be given to the relation of these features to methods of wear testing and not to a detailed discussion of the wearing properties of the different metals used.

LABORATORY AND SERVICE WEAR TESTS

Among the factors found by early investigators to affect the wear of metals are those shown in Table I. These are grouped under two headings: (1) Factors associated with the metals themselves, and (2) factors more directly associated with the external conditions to which the metals are subjected. The importance of each may be expected to vary depending upon the exact conditions encountered. Neglect of one or more of them probably accounts for most of the unsatisfactory or unconvincing data found in published results of wear tests.

Additional evidence of the complexity of the problem of wear testing is secured by comparing results reported by different investigators for similar metals. For this purpose alloy structural steels and tool steels have been selected. In the comparisons shown in Fig. 2 an arbitrary division is made into "metal-to-metal" wear tests, in which the friction is produced between two solid metallic bodies, and "abrasive" wear tests, in which the metal body is worn by a finely divided non-metallic abrasive such as sand, emery, etc. Few comments are required to emphasize that the superiority of one steel over another, as shown in Fig. 2, is in many cases merely a question of which set of tests is considered. Under such conditions, it would seem justifiable to recommend that the study of the wear of metals be carried out under actual service conditions pending the development of more fundamental information relating to laboratory technique. But even this procedure may be accompanied by difficulties, as in the case of the wear of plug gages, recently studied by the author and some of his associates[1] An attempt was first made to secure

TABLE I.—IMPORTANT FACTORS AFFECTING THE WEAR OF METALS

FACTORS ASSOCIATED WITH THE	
METALS THEMSELVES	CONDITIONS OF SERVICE
Metallurgical origin	Material with which the metal is in contact
Chemical composition	Contact pressure
Casting conditions—temperature, chilling effects, etc.	Abrasion speed
Conditions of working—finishing temperatures, direction of rolling, etc.	Condition of surface (smoothness)
Presence of impurities	Presence or absence of lubricants, coolants, film, abrasives, etc.
Grain size	Operating temperature
Constitution and heat treatment	Stress-corrosion effects

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[1] H. J. French and H. K. Herschman, "Wear of Steels with Particular Reference to Plug Gages," *Transactions*, Am. Soc. Steel Treating, Vol. X, No. 5, p. 683, November, 1926.

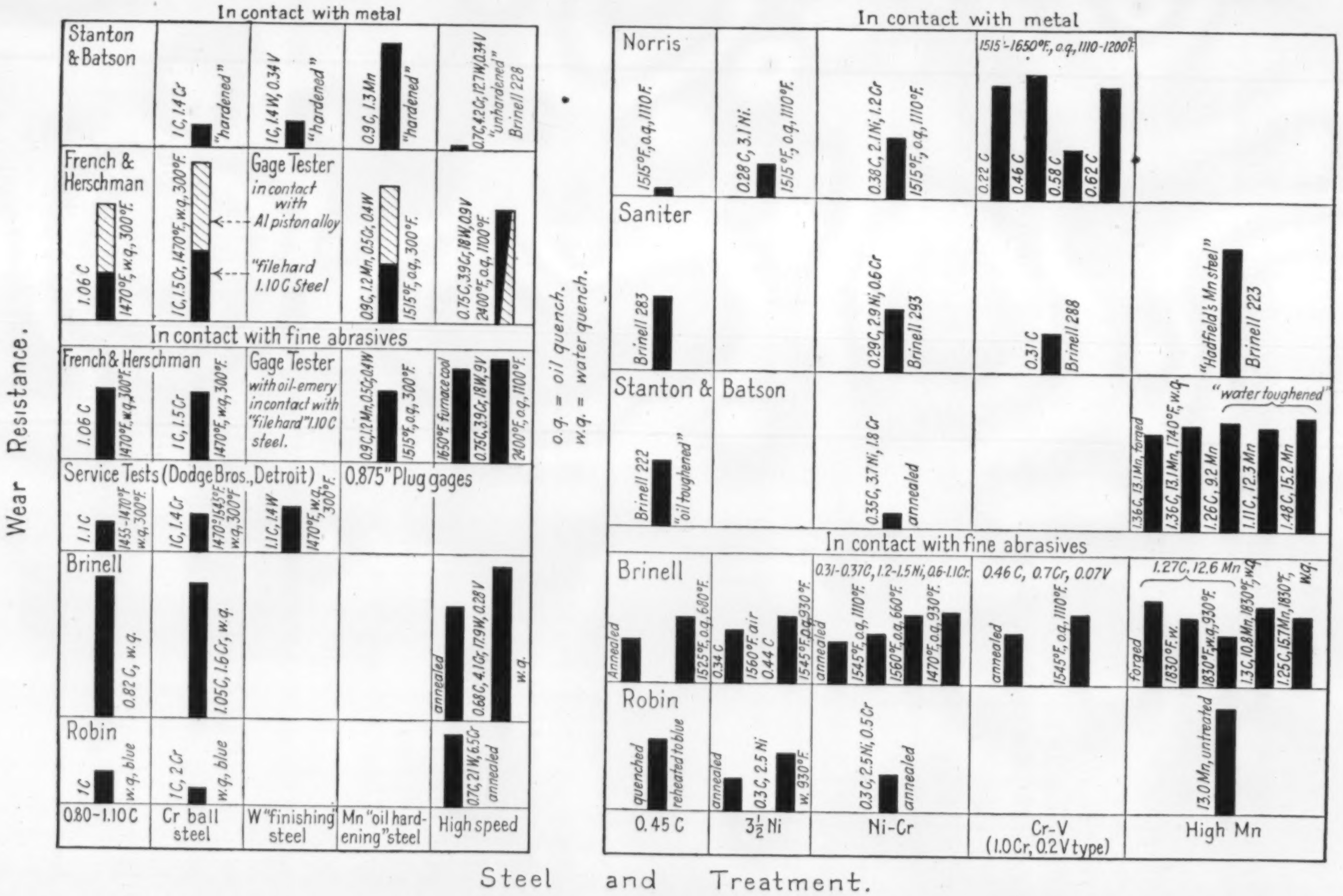

FIG. 2.—"Wearing Properties" of Alloy Structural and Tool Steels as Determined by Various Investigators.

The blocks representing wear resistance determined by different investigators are not all plotted to the same coordinates and have not all been drawn to their respective base lines. Direct comparisons should only be made in individual groups of tests and then only by differences in, and not total heights of, the blocks. For these reasons numerical values are not given. Sources of information are given in the paper by H. J. French and H. K. Herschman, "Wear of Steels with Particular Reference to Plug Gages," *Transactions*, Am. Soc. Steel Treating, Vol. X, No. 5, p. 683, November, 1926.

comparisons in actual service, but the results obtained from a moderate number of gages were not convincing, by themselves, and were suspected of having been affected by variables not readily controlled in ordinary shop practice.

It is not difficult to visualize why this should be so in this case. Parts to be gaged, ordinarily, vary in size and, hence, the clearance between the gage and the work will vary from piece to piece. Some may come to the inspector clean and dry while others may contain abrasives, cutting compounds, etc. Where gages are used by hand, as they generally are, the personal equation may have an important bearing upon gage life. Under such conditions, convincing comparisons can only be secured by a statistical study of the performance of a relatively large number of units.

Notwithstanding these and other difficulties which may be encountered, the answer to a particular wear problem at some one locality can sometimes only be obtained by service wear tests, as it may be impracticable to duplicate in the laboratory the exact conditions which will be encountered in use. On the other hand, there is no assurance that the conclusions drawn will be applicable to what appears to be similar service at another locality.

Where it is practicable to come close to the conditions of service it will generally be possible better to control important variables in the laboratory and results will usually be forthcoming in a shorter time. Furthermore, all carefully controlled laboratory tests add to the fund of knowledge which will some day make it possible to interpret results of wear tests made under one set of conditions into terms of other and quite different conditions. However, to-day it is advisable to consider each application by itself until it can be shown that classification with other applications is justified.

WEAR TESTING MACHINES

At the present time there is no "universal" wear testing machine available and few models on the open market from which to choose. It is, therefore, frequently necessary to construct special equipment for each application and already a host of machines of varied design have been employed or are now, to the author's knowledge, under construction.

It is not considered within the scope of this discussion to take up the advantages of one or another of these different units. However, to illustrate specific applications in some detail, attention will be given to two of the wear testing machines which the author and his associates have used in studying the wear of widely different metal products.

The first of these machines is shown in Fig. 3 and has been used in the study of the wear of plug gages.[1] This machine provides for the automatic raising and lowering of gages in the "work," consisting of split rings, under conditions which permit maintaining constant contact pressure. It simulates the "wringing" action to

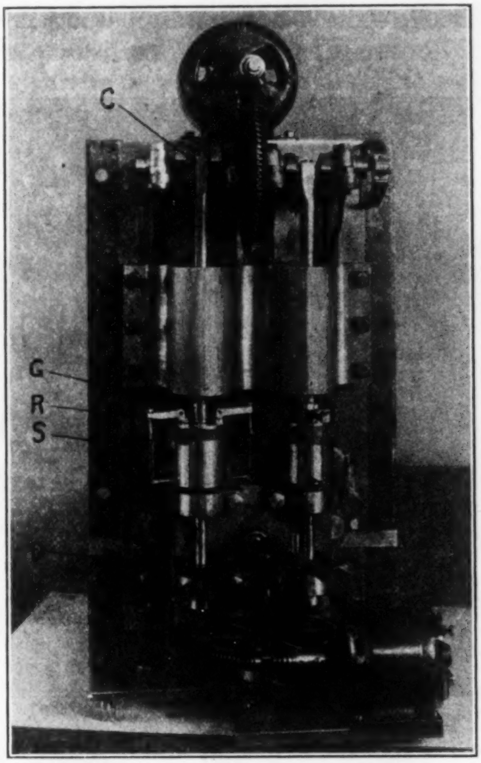

FIG. 3.—Wear Tester Used in the Study of Plug Gages (designed by the author and H. K. Herschman).

G is the gage.
R is the holder for the split rings representing the work to be gaged.
S shows the spring and lever system for maintaining constant contact pressure between the gage and the work.
D is the driving chain for rotating the rings to give the "wringing" action to which plug gages are often subjected in service.
C shows the cranks operating the rods which insert and remove the gage from the work.

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[1] For a detailed description of this machine see H. J. French and H. K. Herschman, "Wear of Steels with Particular Reference to Plug Gages," *Transactions*, Am. Soc. Steel Treating, Vol. X, No. 5, p. 683, November, 1926.

which plug gages are frequently subjected in practice and has given results consistent with practical experience. So far, two types of tests have been made: (1) "metal-to-metal" tests in which the gage rubs directly in contact with the metal of the "work," and (2) "abrasive" wear tests in which the wear is produced by finely divided emery kept in suspension in oil. The wear is measured both by the changes in diameter and in the weight of the gage, and wear resistance is computed on the basis of the number of "gagings" (holes gaged) per unit of diameter decrease in the gage.

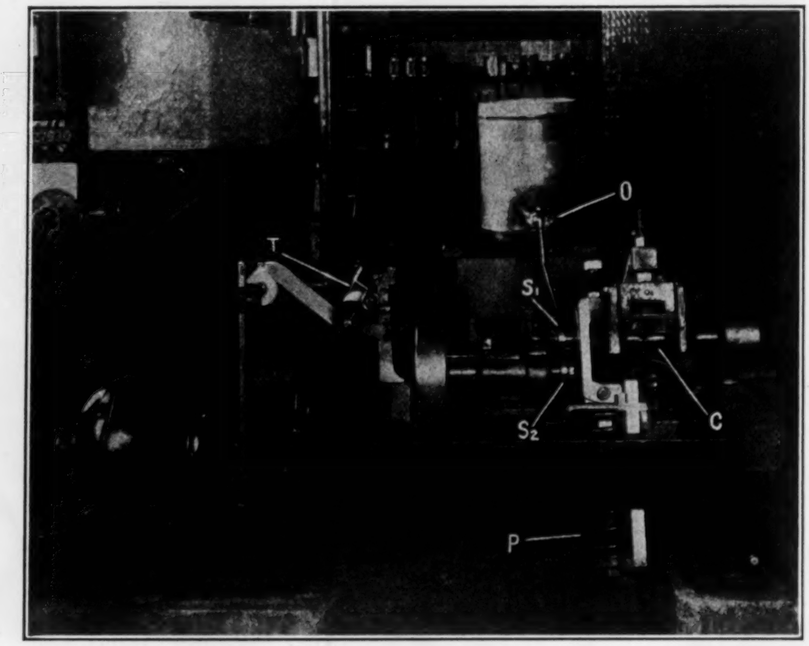

FIG. 4.—Amsler Wear-Testing Machine, Used in the Study of Bearing Bronzes.

S = upper specimen (form of a disk).
S_2 = lower specimen (form of a disk).
T = torque indicator connected to friction dynamometer.
P = spring for controlling contact pressures.
C = cam to give oscillating motion to upper specimen.
O = oil supply for making tests under lubrication.

The second and an entirely different type of wear testing machine is shown in Fig. 4. This is built by Alfred J. Amsler, Schaafhause, Switzerland, and has largely been used by the author and his associates in the study of the wear of railroad bearing bronzes.[1] Essentially this machine provides for the rotation of two disks in contact; the two disks revolve in the same direction but at slightly different speeds so that there is a positive slip between them. Two main driving speeds are provided and the contact pressure may be controlled within a fairly wide range. The machine is further equipped with a friction dynamometer and torque indicator which have been most useful in interpreting the results of wear tests. The machine is further provided with a cam to give the upper specimen a lateral oscillating motion which promotes more uniform distribution of wear. However, such oscillating motion is optional and may be eliminated in any test if desired. Tests may be made "dry" or with lubricants or coolants and, when desired, one of the two specimens may be modified in form and kept stationary so that the other rubs against it in much the same manner as a shaft rotates in a bearing. Wear is determined by weight losses or decrease in diameter of the disks or both.

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[1] For a detailed description of this type of machine see F. P. Hitchcock, "A Universal and Practical Machine, etc., Testing," 1, No. 2, February, 1924, p. 147, Pullman Publishing Co., New York.

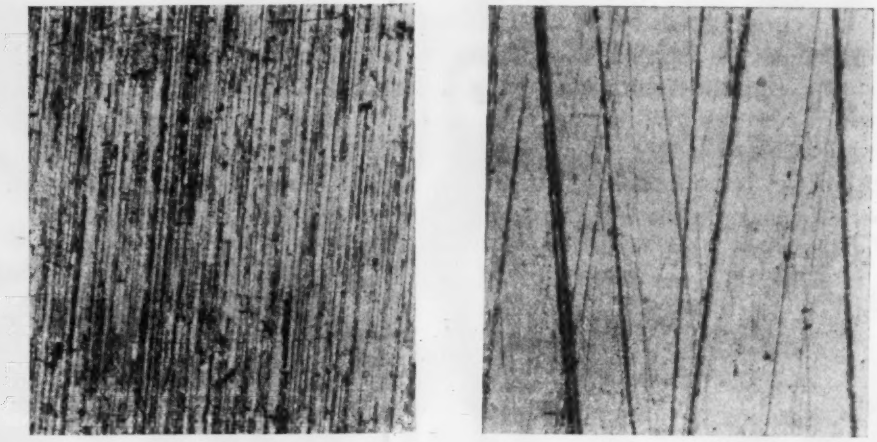
By the addition of a suitable heating furnace (not shown in Fig. 4) tests have been made at temperatures up to about 175° C. (350° F.).

WEAR TEST PROCEDURE

It has been customary in most of the investigations that have been noted to determine weight losses in specimens run for a limited time in a single test. Infrequently a preliminary "run" has been made to "prepare the surface" of the specimen. This has been disregarded and the weight loss in a second but equally short interval is then taken as a measure of the wear. It is only natural to attempt to minimize the time and effort, and hence the cost, of making wear tests, but considerable evidence is now available to demonstrate that such abbreviated tests *may not* give a correct criterion of wear and in many cases are misleading. Two of the important reasons for this will be considered briefly in this discussion and are related respectively to the initial surface of the test specimen and the tendency of metals to "film."

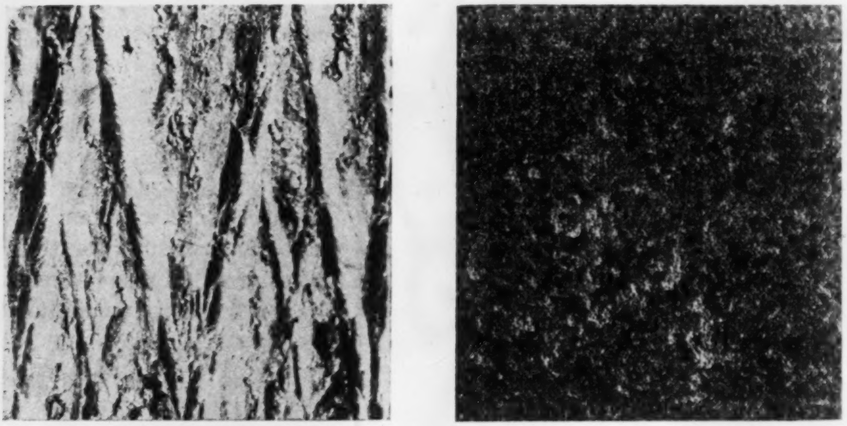
Initial Condition of Surface of Metals:

Ordinarily, wear takes place by the displacement and detachment of metal particles from the surface of the specimens. With two perfectly smooth surfaces in contact the first step in producing wear, penetration or displacement of small particles, is more difficult than when the contact surfaces are rough. From the standpoint of wear and wear testing a ground metal surface is not smooth but rough, as is shown in photograph *A*, Fig. 5. When two such surfaces are moving in contact, some of the projections of the one will, at least momentarily, find the depressions in the other and set up an increase in the resistance to further movement. If the driving force is sufficient to maintain movement the interlocked projections are deformed and if



A—Original ground surface of a copper-tin-lead alloy specimen (× 100).

B—Specimen *A* in contact for 45,000 revolutions with a steel shaft; lubricated with a light oil (× 100).



C—Exceedingly rough type of surface in a lead-free bronze containing about 93 per cent copper and 7 per cent tin after 40,000 revolutions in "dry" tests such as those shown in Fig. 9 (×10).

D—Moderately smooth surface of a bronze containing about 25 per cent lead after about 40,000 revolutions in "dry" tests such as those shown in Fig. 9 (×10).

FIG. 5.—Photographs of the Surface of Bearing Bronzes Before and After Wear Tests Made Under Varying Conditions in the Amsler Wear Tester.

of sufficiently brittle material, may break or be torn off to produce wear. The average contact pressure over the surface does not have to be very high for such effects to be produced, for the small size of the projections and of the particles which are torn off results in a localized stress concentration which can quite readily exceed the "strength" of the metal.

Such localized changes proceed until a new surface is produced which is representative of the metal and the conditions to which it has been subjected. If a typical bearing bronze, originally having a surface like that in photograph *A*, Fig. 5, is run for a long period in contact with a ground-steel specimen in the presence of oil in the Amsler wear tester, under either high or low contact pressures, a smoothing of the surface is observed which at some stage approximates that shown in photograph *B*, Fig. 5. Starting a test with two surfaces of this nature a very low rate of wear is observed, for there are not many projections which can interlock and be torn off. If the bronze and steel are run dry, the surface may become very rough, as is shown in photograph *C*, Fig. 5, or fairly smooth as shown by photograph *D*.

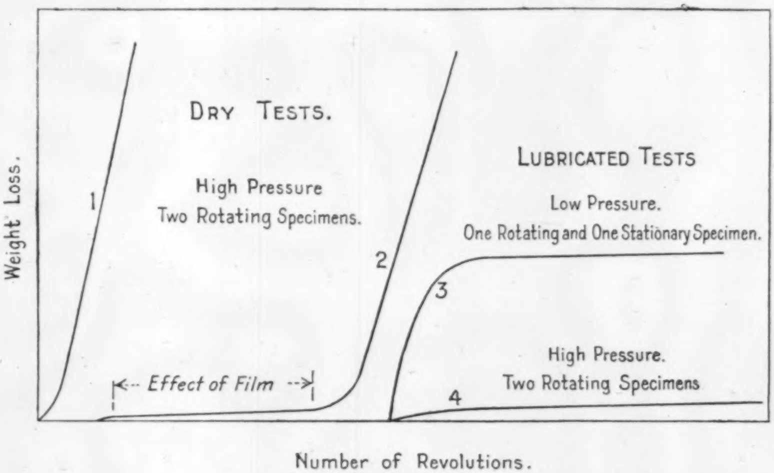


FIG. 6.—Typical Wear-Revolution Curves Obtained in Running Bronzes Against Steels in the Amsler Wear Tester.

Note the effect of film in temporarily reducing the rate of wear as indicated in the body of the chart.

In neither of the cases cited does the original surface closely resemble that produced through frictional wear and it is, therefore, not difficult to understand why the single short wear test, made on a surface finished by one of the usual shop methods, will not necessarily give a correct indication of the wearing characteristics of a metal. Such procedure is often more a test of the original surface finish than of the metal itself.

Expressed in terms of weight losses, that is, wear, the change from surface *A* to *B* and likewise from *A* to *C* or *D*, Fig. 5, takes place as shown in Fig. 6. In the case of the "dry" wear tests the relation between the rate of wear in the first and subsequent intervals is partly a question of the relative roughness of the original surface and that which exists after appreciable wear has taken place. The weight-loss-revolution curve may, therefore, approximate a straight line or the wear rate in the first interval may be higher or lower than is represented by the slope of this line. In curves 1 and 2, Fig. 6, the initial rate of wear is lower than that in subsequent intervals. This has been the effect which has usually been observed in the copper-tin-lead alloys prepared with an original surface like that shown in photograph *A*, Fig. 5. However, this change in rate of wear is not wholly a function of the initial condition of the surface, for it has been found that bronzes cast in metal molds seldom show the same degree of change due to the initial surface as do the same alloys cast in sand molds. The former "wear-in" more quickly and then wear faster.

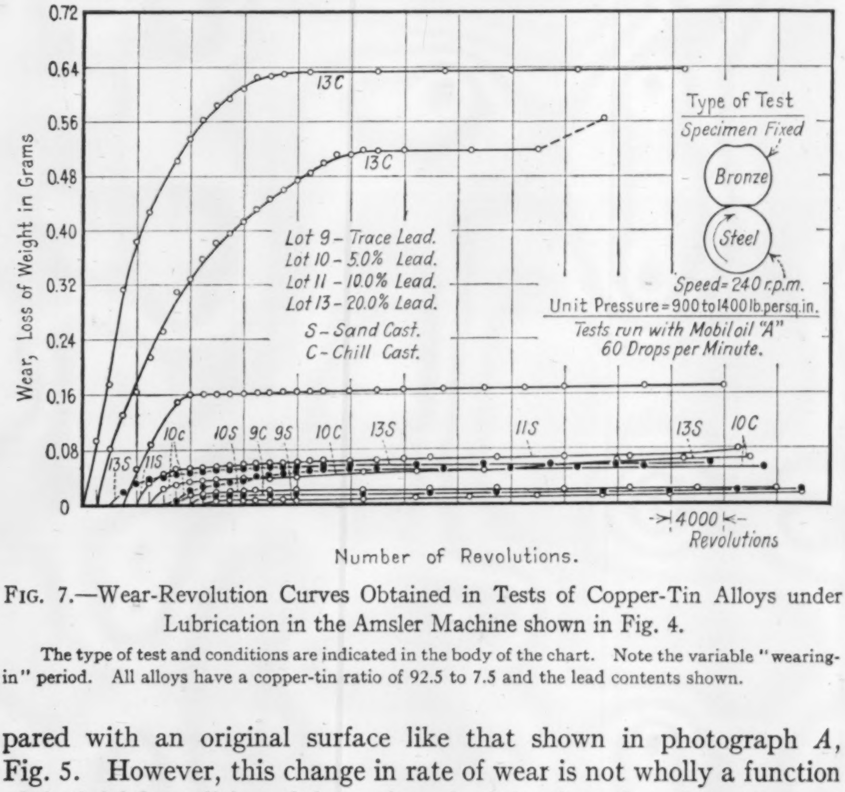


FIG. 7.—Wear-Revolution Curves Obtained in Tests of Copper-Tin Alloys under Lubrication in the Amsler Machine shown in Fig. 4.

The type of test and conditions are indicated in the body of the chart. Note the variable "wearing-in" period. All alloys have a copper-tin ratio of 92.5 to 7.5 and the lead contents shown.

When the same alloys are run at either high or moderate contact pressures in the presence of oil, the wear in the early stages is very much greater than at subsequent intervals; and finally, when the surface has assumed a condition which looks like that of photograph *B*, Fig. 5, the rate of wear is at a very low value which, from the standpoint of reproducible wear tests, may be considered practically nil. This is shown by curves 3 and 4 in Fig. 6.

To illustrate this from actual experiments there are summarized in Fig. 7 the wear-revolution curves for a number of copper-tin alloys containing different proportions of lead, in which specimens were taken from "sticks" cast in sand and likewise in metal molds (chill-cast). These results indicate that after an initial period, which may vary widely in duration and amount of wear, the various alloys practically stop wearing so long as lubrication is maintained; at least the rate of wear assumes an entirely different order of magnitude which is so low that it must, in the light of present-day wear test methods, be considered negligible. It would, therefore, seem necessary also to make unlubricated wear tests to differentiate between the different alloys, for, according to Fig. 7, so long as lubrication is maintained the wear subsequent to an initial "wearing-in" period may be neglected. Practically, it is as important to know the behavior after "wearing in" as it is to know the behavior during this initial period.

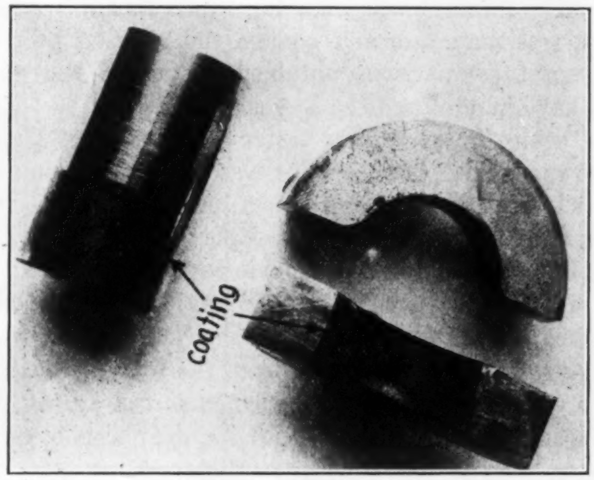


FIG. 8.—"Film" Formed on Gages and Rings when Tested "Dry" in the Gage Wear Tester shown in Fig. 3.

The case of bearing bronzes is not the only one that can be cited in support of the importance of initial surface condition of specimens in relation to the results obtained in wear tests. Another case is that of precision plug gages. It is now more or less generally recognized that for close tolerance work of the order of a few ten-thousandths of an inch, the life of a gage is partly, and in some cases quite largely, dependent upon the smoothness of the original finish. The wear resistance of the metal is probably a larger factor where larger tolerances are permitted.

There are, of course, conditions where the original surface is not so important and among these are the cases in which wear is brought about by particles having a relatively high cutting or penetrating power in comparison with the resistance to penetration of the metals, such, for example, as wear by sand, rock, emery, etc., previously called "abrasive wear."

"Filming" of Metals:

One of the important features sometimes encountered in wear tests, which has so far received comparatively little publicity, is "filming." The formation of a coating which was not present at the start of the test may radically change the wearing properties and completely upset comparisons obtained in one of the abbreviated tests which have heretofore been so generally used.

In first using the gage wear tester shown in Fig. 3 an attempt was made to run "dry," but a coating was soon formed on the contact surfaces which tended to cut down wear, produced erratic results and caused the gage and ring to "seize." This film, an example of which is shown in Fig. 8, looked like iron oxide and was ascribed to the fact that the small metallic particles torn from the contact surfaces were pyrophoric, that is, the particles were of such small mass in proportion to the energy required to tear them from the body of the metal that they burned or at least oxidized and when forced against the metal formed a coating or film. Carrying out the wear tests in the presence of water decreased the difficulties from such coatings.

A more important illustration of "filming" has been found in the "dry" wear tests of bearing bronzes in the Amsler machine. The composition of the films produced is not known with certainty, but they have apparently been of two types. The first of these, comprising films which look like copper oxide, have occasionally been observed in the copper-tin alloys which are practically free from lead. These same films also have occurred in the high-leaded bronzes, but they are not so easy to see in the latter alloys due to the more frequent formation of a second type of coating, a black film which is either lead or a lead compound. Tests so far made, including X-ray analysis, have failed to reveal its exact nature. The conditions favoring formation and control of such films are not yet known but they have occurred most often in alloys containing around 15 to 25 per cent of lead, cast in sand molds.

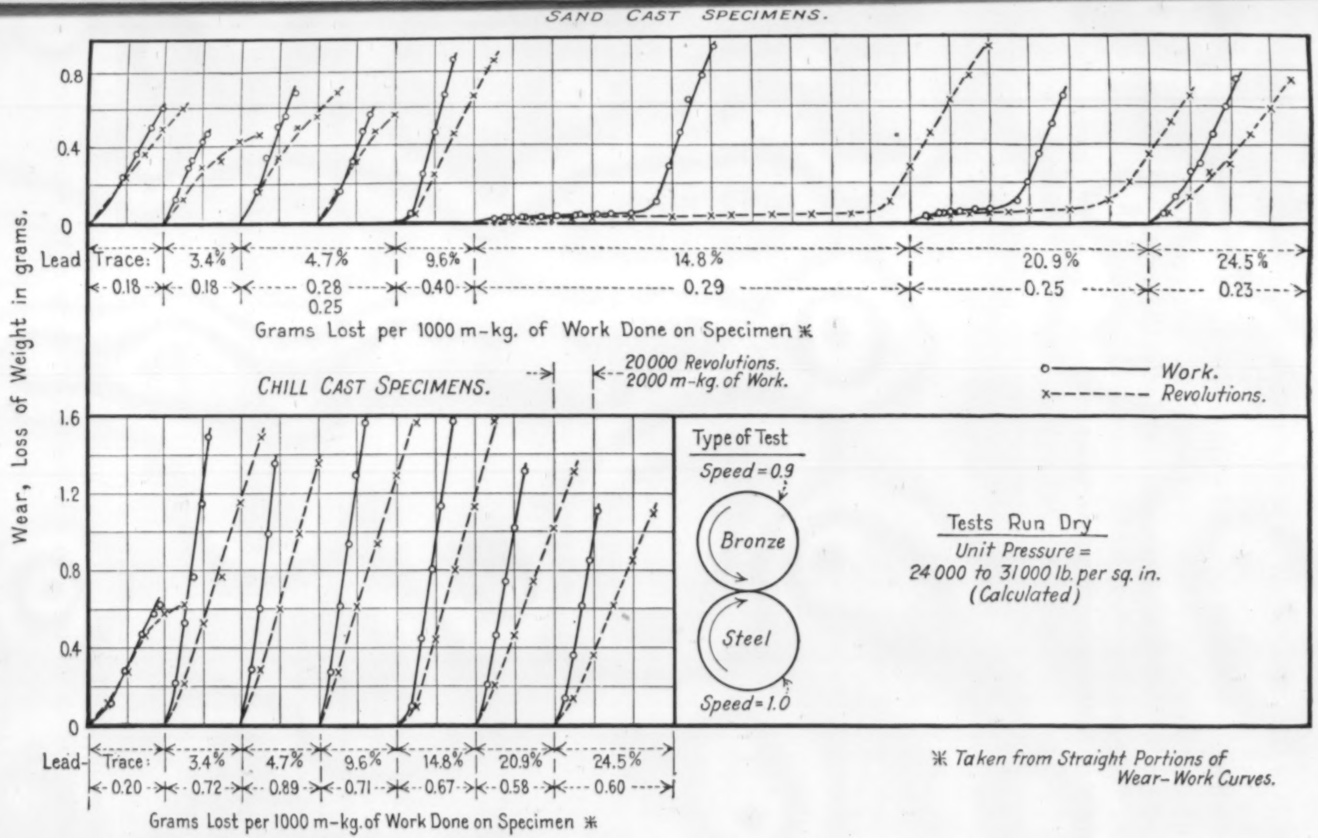


FIG. 9.—"Dry" Wear Tests of Bronzes on the Amsler Machine in Which the Effect of Film Is Shown by a Temporary Decrease in the Rate of Wear.

The effect of "film" can be seen in the wear-revolution and wear-work curves for the sand-cast bronzes containing respectively 14.8 and 20.9 per cent lead, in that the first portions of these curves are nearly horizontal and later turn upward to approximate the slopes found in the other alloys. This more or less rapid change in slope is generally concomitant with the breakdown or disappearance of the "film."

Such films exert a very marked effect on the "bearing properties" and wear of the bronzes. With the formation of film there is a variation, and frequently a noticeable drop in the torque, and the wear drops to very low values as will be seen from examination of Fig. 9 for sand-cast specimens containing around 15 to 20 per cent of lead. When the wear test has been continued for sufficiently long periods, this film has generally, though not always, broken down with an accompanying change (frequently an increase) in the torque and an increase in the rate of wear. The time of formation, duration of existence and the breakdown have varied not only in different alloys but in duplicate specimens taken from the same casting and tested under what were believed to be like conditions. A photograph of the film to which reference is made is shown in Fig. 10 for three sand-cast bronzes containing different proportions of lead. Were this in natural colors it would be somewhat easier to see that the intensity of the black color is greater in the alloys containing 20 to 25 per cent of lead than in that containing 15 per cent.

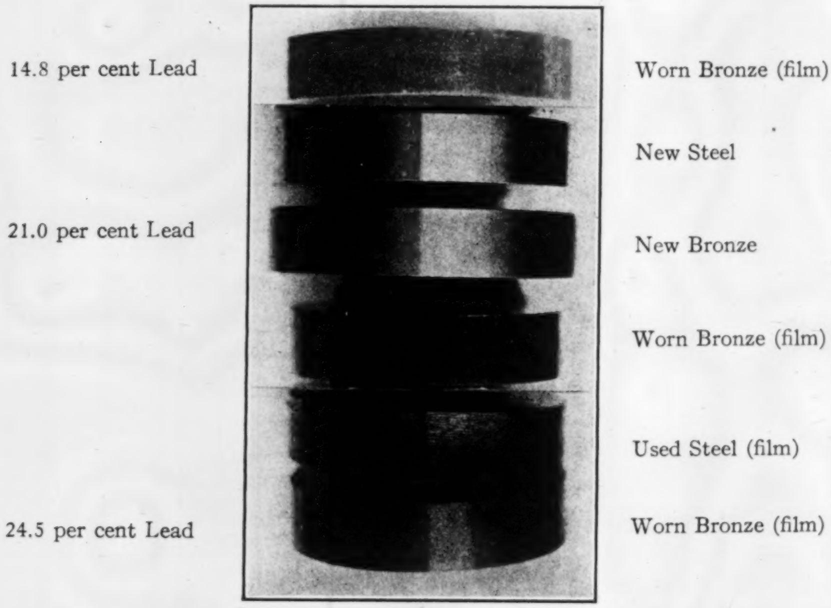


FIG. 10.—Photograph of the Films Formed in Bearing Bronzes Containing Different Proportions of Lead.

The alloys contain copper and tin in the ratio of 92.5 to 7.5, lead as indicated. The films shown are those present after 5,000 to 10,000 revolutions in the Amsler tester under conditions similar to those represented by tests recorded in Fig. 9.

The described effects constitute another reason why great care must be used in the interpretation of short-time wear tests, why studies should be made under conditions approximating as closely as possible those to which the metals will subsequently be subjected in service, and why specimens must frequently be subjected to friction for long periods to obtain a reasonably correct idea of their wearing properties. There are other important reasons than those given, but the majority have received somewhat more attention and are better recognized than those which have been considered in this discussion.

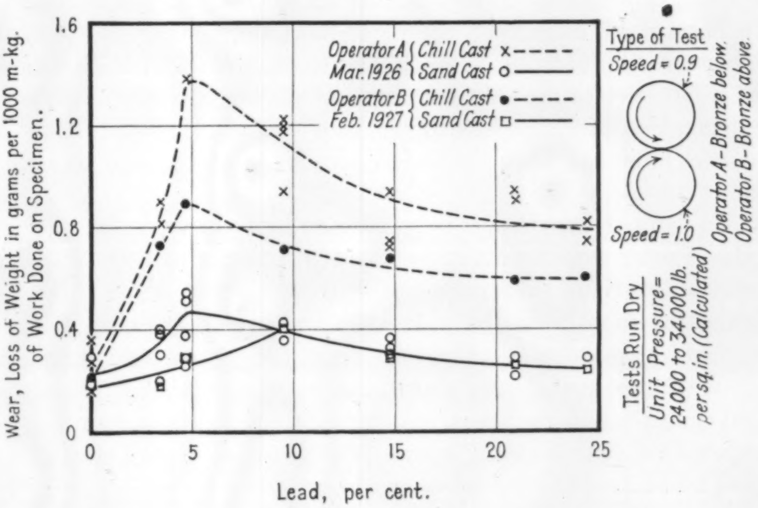


FIG. 11.—Effect of Lead on the Wear of Bearing Bronzes as Determined in Two Sets of "Dry" Wear Tests in the Amsler Machine, at Room Temperature, Made about a Year Apart by Two Different Operators.

Cited to show the possibility of reproducing results when wear tests are carefully made. See text of report for discussion. Details of the tests are given in the body of the chart.

REPRODUCIBLE WEAR TESTS

The emphasis that has been placed on the need for care in making wear tests and the interpretation of results, as well as the complexity of the problem of wear testing, is not to be taken as indicating that reproducible wear tests cannot be made nor that results cannot be obtained in the laboratory which will be consistent with practical experience.

The ordinary practice in making Amsler wear tests has been to measure weight losses in a number of successive runs, each of 10,000 revolutions, and subsequently to plot the weight loss for each run against the frictional work. Subsequent to a "wearing-in" period and providing no unusual circumstances arise, the wear-work curves have been found to be straight lines and the slope of this line expressed in grams per kilogram-meter (wear per unit of work) has been taken as a measure of the wear rate of the metal. In all "dry" wear tests on the Amsler machine, to which reference is made in this discussion, copper "scrapers" have been used, that is, copper strips have been so placed on the machine that they continually bear against the rotating specimens. This was originally used in an attempt to overcome difficulties with film formation by removing particles torn from the surface of the specimens almost as soon as they were detached. The "scrapers" have been shown to have no measurable effect upon the wear of the bronzes or steels here considered and since originally used it has been found that they are not always effective in preventing the formation of films. However, they probably assist in this direction and have not been discarded pending a more detailed study of their effectiveness.

To demonstrate that it is possible to reproduce results, some tests in the Amsler machine may be cited. A series of tests on bronzes containing varying proportions of lead, was made during March and neighboring months in the year 1926 and are recorded in Fig. 11 as obtained by operator A. The changes in the rate of wear with change in lead content are clearly shown, as is the fact that bronzes cast in metal molds (chill-cast) wear much more rapidly than bronzes of like compositions cast in sand molds (sand-cast). About a year later, during February, 1927, a set of specimens from the same castings (not necessarily the same disks) was turned down to somewhat smaller diameter and a new operator, designated as operator B in Fig. 11, who had had a limited experience with the Amsler wear tester but who took no part in the first set of experiments, was assigned the work of making a complete set of wear tests comparable to the original. However, he was instructed to interchange the position of the bronze and steel specimens so that instead of having the bronzes on the lower shaft of the machine, as in the first set of tests (see Fig. 4) they would be on the upper shaft.

The results obtained are given in Fig. 11 for direct comparison with those obtained in the first set of tests made about a year earlier. Despite the change in position of the specimens, the use of different disks from the various castings and a change in the operator, very close checks were obtained. The lower rate of wear of the chill-cast bronzes in the second set of tests is due to the fact that smaller disks were used. The wearing surfaces of the disks were, therefore, further from the metal which had been chilled to the greatest degree and hence wore less rapidly. The difference in the wear rates in the first and second sets of tests for the sand-cast alloys containing about 2.5 to 10 per cent lead is likewise due to a variation in the degree of chill of the metal. In one case a sand-cast specimen which gave a relatively high rate of wear under conditions comparable to those of the first set of tests, was turned down and then gave a lower wear rate which checked the value obtained on the smaller disks used in the second set of wear tests.

CHECKING SERVICE WEAR TESTS IN THE LABORATORY

As an illustration of the possibility of getting in the laboratory results consistent with practical experience and service wear tests, reference can be made to the results of investigations with the gage tester, Fig. 3, which have already been published in detail.[1]

SUMMARY

The subject of wear testing of metals is too broad to give much detailed information within the allotted space. It has, therefore, been necessary to restrict comments to a few of the variables encountered; others, equally and perhaps more important, have had to be neglected.

However, this paper is not intended as a record of experimental work but has been written to point out a number of important features connected with wear testing of metals which have so far not been given a great deal of attention.

The features which should be emphasized are first, that prior experience indicates that the wear testing of metals is not such a simple matter that abbreviated tests, carried out in a few minutes, can be relied upon to give a correct indication of wearing properties. Two features cited, which may interfere with successfully carrying out such tests, are the importance of the initial condition of the surface of metals and the tendency of metals to "film."

Secondly, until more fundamental information becomes available each application of metals involving wear must be considered by itself. Tests should be made under conditions approximating as closely as possible those to which the metals will be subjected in service and because of this there is now no single testing machine which can be said to be universally applicable.

Thirdly, difficulties may be encountered in carrying out service wear tests just as they are encountered in making wear tests in the laboratory but, for the present, whenever possible, laboratory and service wear tests should be carried out side by side.

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[1] H. J. French and H. K. Herschman, "Wear of Steels with Particular Reference to Plug Gages," *Transactions*, Am. Soc. Steel Treating, Vol. X, No. 5, p. 683, November, 1926.

Where care and patience are observed it has been found possible to duplicate wear test data, and cases are cited where it has been possible in the laboratory to obtain results consistent with practical experience.

Acknowledgments.—The author acknowledges the assistance of a number of his associates in the procurement of the experimental data included in this paper and for suggestions which have helped in the development of wear test methods. Among these are W. LeC. Harbaugh, Research Associate at the Bureau of Standards from the Magnus Metal Company, Chicago, Ill., working on bronzes; H. K. Herschman, Assistant Metallurgist, Bureau of Standards, working largely on gage steels; S. J. Rosenberg, Assistant Metallurgist, and R. F. Staubly, Jr., Laboratory Aid, Bureau of Standards, for assistance in various phases of the work.

DISCUSSION

THE PRESIDENT (*Mr. J. H. Gibboney*[1]).—I am sure that you will have already concluded from my remarks in opening this annual meeting that I am deeply interested in the matter of wear testing and recognize in such a field of work a splendid opportunity for the testing engineer to render a large service to industry. I wish to take this opportunity to commend very highly the missionary work that the Bureau of Standards is doing in directing attention to this important economic problem and the fine cooperation it has had from the Magnus Company which has made the present researches on bearing metals possible. I hope to see this work go on. It is a privilege our great industries have, to help in a broad and unselfish way to solve these difficult service problems by careful researches, and such evidences as these are convincing of our hopes in producing materials that will give increasing better service. **Mr. Gibboney.**

In any test of the resistance of a metal to wear, whether it be a laboratory test or a service test, the results are certain to be affected by variables, some of which are very difficult, if not at times impossible, to control. Such variables may consist of inherent characteristics of the metal itself or they may be induced by external factors associated with conditions of service.

Some of these variables may be easily recognized and allowed for in the computation of results, while others are more subtle and are likely to introduce mystifying errors, the origin of which can be no more than guessed at. Of course, the recognition and control of such variables is a much more simple process in case of laboratory tests than of service tests; in fact in the latter type of test, even when they are easily recognizable they can often be allowed for only in the very broadest sense—such as allowances made for differences in summer and winter temperatures, for differences in weight or speed between two classes of locomotives, or between two locomotives of the same class performing different types of service, for differences in quality of various lubricants used, etc.

Service tests, however, are by their very nature rough-and-ready affairs as compared with laboratory tests and, therefore, broad allowances for the former will bear about the same relation to the finer

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[1] Chief Chemist, Norfolk and Western Railway Co., Roanoke, Va.

Mr. Gibboney. allowances of the latter as will the somewhat broad results to be expected bear toward the finer-drawn results of the laboratory test.

The chief benefit to be expected from the laboratory test is that it will point the way for experimental work in any effort toward securing or producing a bearing material with the best possible wearing qualities. As for its usefulness as a guide in any final decision concerning relative qualities of two bearing materials, such evidence should be accepted subject to confirmation by an actual service test, for service itself is the final test for any material, and the first and last objective of any test is to determine relative fitness for service. While a laboratory test may safely be expected to furnish a fairly accurate index as to what may be expected from a service test, still it may fall down entirely at times, since we can never be perfectly sure of duplicating actual service conditions in the laboratory. Therefore, after the laboratory test has pointed the way, the service test should always be resorted to for confirmation.

Mr. Corse. MR. W. M. CORSE[1] (*presented in written form*).—The author has given a very interesting picture of an important project now being studied at the Bureau of Standards. A careful search of the literature and conversations with prominent testing engineers, both in America and abroad, who are interested in this subject shows that this is practically a pioneer investigation when one considers the fact that it is probably the first time that reproducible results and information consistent with practical experience have been secured in the laboratory.

It is a fact generally conceded by investigators of this subject that wear commences only when there is metal to metal contact. If this be true, then the presence of a lubricant in a continuous film must mean the absence of wear. The logical conclusion to draw from this statement is, of course, that the lubricant in a series of tests means undue prolongation of the time as well as the introduction of a variable which is very difficult to control. It is always desirable to eliminate, where possible, any variables of this kind in testing operations.

It was decided to make these bearing tests described on dry specimens at first. In the usual type of machine, this is not practicable because the friction of the specimens raises them to a temperature which vitiates results. In the Amsler machine, the contact is a line or, if elasticity is taken into account, a narrow surface. This means that contact pressures can be raised to fairly large amounts without any heating of the specimens. In these tests, the Bureau of Standards

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[1] Newell, Corse and McDaniel, Washington, D. C.

Mr. Corse. has succeeded in calculating these line contact pressures and they were found to run into figures of considerable magnitude. In all cases, the dry specimens ran relatively cool. Some engineers claim that lubrication is absolutely essential to a test that will give results comparable with practice, but as stated above the practicable difficulties are so great that up to date no authentic tests have been published on wear testing of this kind.

While as the author states in his paper the subject of wear testing of metals is too broad to give much detailed information within the space of the paper, it is evident several very important fundamental facts have been developed. The preliminary work reported by the author is so encouraging that there seems little doubt about the ultimate success of a program which can easily extend over a period of several years.

Mr. Shoemaker. MR. R. J. SHOEMAKER[1] (*by letter*).—The method of wear testing described by Mr. French is of particular value in the determination of the relative wear resistance of bronze bearings of different compositions used in railway service. This is of especial importance at the present time on account of the claims made by certain manufacturers that chill-mold castings are superior in wear resistance to those cast in sand.

With reference to the curves of Figs. 7 and 9 of the paper, the chill-cast specimens showed a much greater rate of wear than the corresponding sand castings. Furthermore, there was greater resistance to wear in the mixtures containing from 10 to 15 per cent of lead than in those where there was either an excess or a deficiency of lead. The rate of wear during the wearing-in period likewise was less for sand than for chill-cast metal. The same differences in wear resistance of chill and sand-cast specimens as brought out in Mr. French's paper are indicated by service tests of bronze locomotive bearings used in railway service. One of the large railway systems of the country has recently finished a very exhaustive test to determine the relative merits of sand and chill-cast driving brasses. The results indicate that sand castings are superior to chill castings. Furthermore, there was a very marked difference in favor of the sand castings as regards the amount of lubrication used.

Without going into a detailed discussion of this subject it may be stated that the primary reason for inferior wear resistance of chill castings is the absence of the relatively hard copper-tin constituent in a mixture which is suddenly chilled from a high temperature. A chill casting lacks the essentials of a true anti-friction metal, namely,

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[1] Engineer of Tests, Magnus Co., Inc., Chicago, Ill.

Mr. Shoemaker. a relatively hard wear resisting substance contained in a soft plastic matrix. This is further indicated by the high degree of brittleness of chill castings. The relatively open-grain structure of a sand casting has a tendency to hold lubrication to a greater degree than a chill casting due to the dense close grain and homogeneous structure of the latter. The effect of structure on anti-friction quality of bronze is clearly shown in a paper by Francis W. Rowe, presented before the British Institute of Metals.[1] Mr. Rowe states: "It is fairly certain that the essential for a bronze alloy to resist frictional wear and give a low coefficient of friction is a network of hard particles in a comparatively soft and plastic matrix, and that anything which tends to homogenize the mass and decrease the difference in hardness between the two components increases the friction and wear."

The method of wear testing as developed by Mr. French and his associates at the Bureau of Standards is a very valuable contribution to the science, as it gives an accurate means of determining the effect of variations in composition and structure on the wear resistance of metals.

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[1] F. W. Rowe, "Physical Properties of a High-Tin Bronze," *Proceedings*, British Inst. of Metals, Vol. 32, No. 2, p. 78.

METHODS FOR DETERMINING THE TENSILE PROPERTIES OF THIN SHEET METALS

By R. L. Templin[1]

Synopsis

The paper describes methods for determining the tensile properties of thin sheet metals varying in thickness from 0.005 to 0.250 in., inclusive. The methods discussed have been used for a number of years in the laboratories of the Aluminum Co. of America, not only for aluminum and magnesium and their alloys but also for numerous other sheet metals both ferrous and non-ferrous.

Economical methods for the machining of tension test specimens, application of gage marks and the measuring of specimen dimensions are discussed. Proper methods for gripping specimens, types of testing machines, testing speeds and a method for determining "yield point" are described in considerable detail.

The character of the test results obtained using the methods described is discussed, together with the treatment of such data.

An attempt is made to show that when the methods described are used, large numbers of tension test specimens taken from thin sheet metals can be tested at a minimum cost with very satisfactory results. A brief discussion is given of the commercial application of tension test data to research, fabrication, inspection and specification of thin sheet metal products.

The Tension Test

The merits of the tension test as applied to metals have long been recognized but the application of this test to thin sheet metals presents certain difficulties which do not obtain, at least to such an appreciable extent, when testing metals in the larger sections. Recognizing these difficulties, many attempts have been made with varying degrees of success to substitute other tests such as bending, cupping and hardness for the tension test. Unfortunately, such other tests quite frequently give but little information, are susceptible to considerable errors and often are not applicable throughout wide ranges of kind, temper or thickness of material.

Appreciating these factors and with the idea that the tension test would be without most of the limitations just indicated, a serious attempt has been made in the laboratories of the Aluminum Co. of America to develop suitable methods and equipment for making tension tests of thin sheet metals, with quite satisfactory results. The various kinds of sheet metals to which we have applied this testing procedure include aluminum and its alloys, steel, brass, copper, nickel, zinc, monel metal, phosphor bronze, tin plate, magnesium and nickel silver in numerous gages and tempers. These methods and equipment have been approved or adopted in whole or in part by many other firms and certain government departments with apparently equally satisfactory results, so it seems worth while presenting them before this Society for its consideration and criticism.

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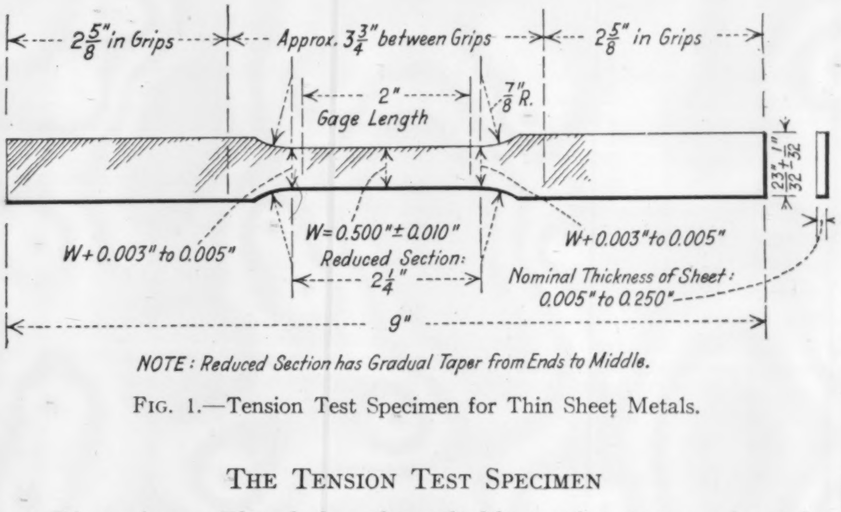
[1] Chief Engineer of Tests, Aluminum Co. of America, New Kensington, Pa.

An attempt will be made to point out that when a certain tension test specimen is used together with suitable test procedure and apparatus, very satisfactory results on thin sheet metals can be obtained readily and economically. Under such conditions, the scope and value of the tension test is materially increased.


$2\frac{5}{8}$" in Grips
Approx. $3\frac{3}{4}$" between Grips
$2\frac{5}{8}$" in Grips
2"
Gage Length
$\frac{7}{8}$" R.
$\frac{23}{32}$" ± $\frac{1}{32}$"
W + 0.003" to 0.005"
W = 0.500" ± 0.010"
Reduced Section:
$2\frac{1}{4}$"
W + 0.003" to 0.005"
Nominal Thickness of Sheet:
0.005" to 0.250"
9"

NOTE: Reduced Section has Gradual Taper from Ends to Middle.

FIG. 1.—Tension Test Specimen for Thin Sheet Metals.

THE TENSION TEST SPECIMEN

Dimensions.—The choice of a suitable tension test specimen for thin sheet metals varying in nominal thickness from 0.005 to 0.250 in. involves a consideration of many factors which have been quite thoroughly discussed by a Section on Thin Sheet Metals, of the Society's Committee E-1 on Methods of Testing, in its report[1] of last year. Additional considerations affecting such a choice together with the results of an investigation on the effects of size and shape of tension test specimens on the tensile properties of thin sheet metals have been presented by the author previously.[2] The dimensions of the tension test specimen referred to in the discussion which follows are shown in Fig. 1. This specimen, it may be noted, conforms to the requirements indicated in the proposed standard specimen for thin sheet metals now before this Society.[1]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 531 (1926).

[2] R. L. Templin, "Effects of Size and Shape of Test Specimen on the Tensile Properties of Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 378 (1926).

Specimen Blanks.—Specimens of the dimensions indicated are machined from specimen blanks which in turn are sheared or sawed from properly identified samples of thin sheet metals. These samples are in nearly every case of sufficient size to permit of more than one specimen blank being cut therefrom. In many cases the samples permit of blanks being cut from them both parallel and normal to the direction of rolling of the material. The specimen blanks are cut from samples as recommended in the current report referred to above. The numbers corresponding to order, lot, heat or item of material which appear on the samples are transferred to the specimen blanks as they are prepared. In the case of aluminum and many other metals, it has been found quite satisfactory to simply scratch the identification numbers on the blanks, using a hardened steel scriber. This procedure is much to be preferred in the case of the thinner sheet because the use of steel dies or stencils causes sufficient deformation in the ends of the specimens to prevent their being properly gripped during testing. In the case of the thicker gages, however, the steel dies or stencils can be used without affecting the test results, depending on the kind or temper of material and the method used in stenciling.

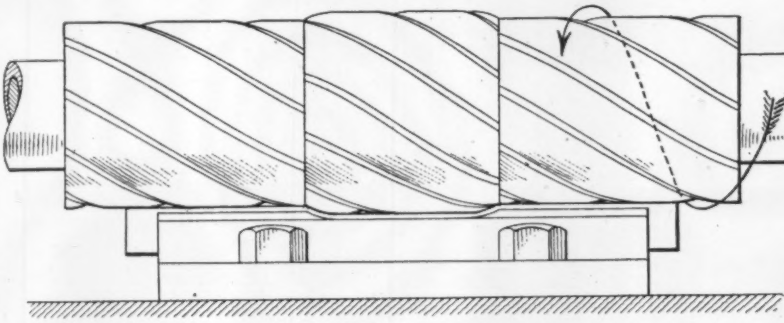

FIG. 2.—Sketch Showing Milling Procedure for Machining Thin Sheet Tension Test Specimens.

We have found for material about 0.1 in. thick or less that the specimen blanks can be sheared from the samples using a suitable punch and die in either a foot-power or motor-driven single-action press, with an appreciable saving in time and cost. Best results are obtained by using a punch and die arranged to cut on three sides only of the specimen blank, with a spacing device to insure that the blanks will be of the desired width. When using such a procedure, there is a minimum waste of material permitting a maximum number of blanks to be cut from a given sample.

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[1] See Report of Committee E-1 on Methods of Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I, p. 584 (1927).

Machining.—With the specimen blanks prepared as just indicated, the problem of accurately and economically machining the test specimens from them must then be considered. As many will appreciate, the machining operation may be expensive, especially if the machining is done with a milling machine in a direction parallel to the longitudinal axes of the specimens. In our early attempts to solve this machining problem it became evident that a very marked reduction in cost would be realized if the specimens were milled transversely. Accordingly, a jig for holding the specimens was designed and made for use in an ordinary milling machine and a special profile cutter was provided so that specimens corresponding approximately to the dimensions indicated in Fig. 1 could be machined in multiple by transverse milling as indicated in Fig. 2. Before finally adopting this milling procedure, however, an investigation was made covering various grades and tempers of sheet metals, for the purpose of comparing the effects on the mechanical properties due to milling specimens longitudinally and transversely. The results of this work indicated

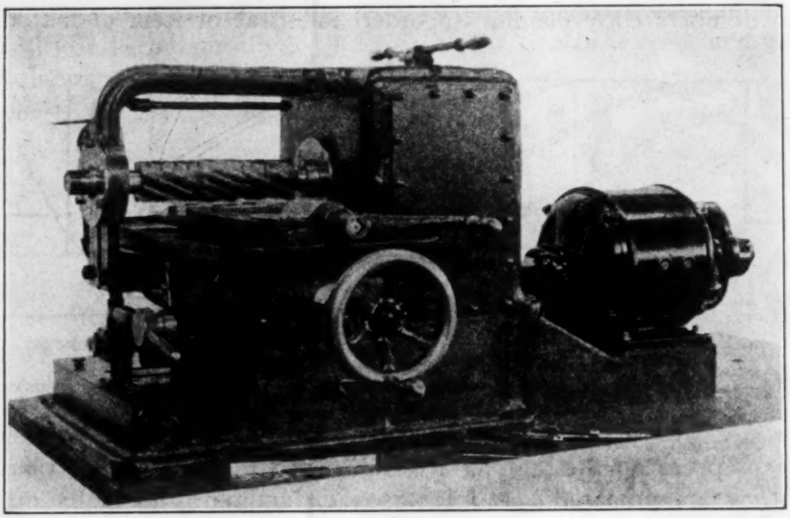

FIG. 3.—Thin Sheet Metal Tension Test Specimen Milling Machine.

quite clearly that, provided the cutters were kept reasonably sharp, any differences in the mechanical properties of the materials due to the direction of machining were quite negligible.

As the volume of the testing work and the number of our testing laboratories increased it became advisable to provide special milling machines designed for the sole purpose of milling tension test specimens from thin sheet metals. Accordingly, the milling machine shown in Fig. 3 was designed and built. Such milling machines are now in use in each of our sheet mill laboratories and have given satisfactory service for the past three to four years. For aluminum and its alloys, a special profile milling cutter 3.25 in. in diameter by 3 in. long is used, together with two stock end cutters 3 in. in diameter by 4 in. long, on a 1.25-in. diameter arbor running at a speed of 240 r. p. m. Under such conditions one of these milling machines will finish B. & S. gage No. 24 (0.020 in. thick) test specimens at the rate of ten per minute.

The specimens are machined in multiple by using a pack of specimen blanks from 2 to 3 in. thick. This pack is placed in the vise of the milling machine, securely clamped, then one cut made across the pack. Screw clamps are next applied to the ends of the pack close to the vise, after which the pack is turned over (using the clamps for centering) and a second final cut taken. In some cases, usually when the specimen blanks are over-width, the pack is fed through again, after each of the cuts just noted to remove any effects due to spring in the arbor of the milling machine. Certain kinds and tempers of sheet metal specimens will have a burr on their unsupported edges resulting from the milling operation and unless extreme care is taken in removing these burrs, the two end or outer test specimens may be damaged. In such cases an extra blank may be placed on each side of the pack and discarded after machining. By using these extra blanks the spoiling of test specimens from the cause just indicated is eliminated.

The milling cutter shown in Fig. 2 was assembled from two standard and a special cutter, all being of the spiral, coarse tooth, high-speed steel type. Experience has shown that it is nearly always necessary to regrind stock milling cutters to make sure they are of the same diameter, within the desired tolerance of ±0.0005 in. After being reground, such cutters are used in matched pairs. In order properly to regrind the special profile or central cutter, we found it advisable to design and build two special grinding fixtures, one for grinding the crown of the central portion and the other for grinding the radii on the ends of the cutter teeth. With these grinding fixtures, the profile cutters can be reground for a nominal cost. A second or spare set of cutters is regularly provided in each laboratory.

Marking and Measuring.—After machining, the specimens are ready to receive the gage marks and be measured. Both of these operations are done by the man making the tests and not by a machinist. We have designed and used a gage marker for quickly and accurately placing the gage marks on the specimens. This marker is shown in Fig. 4, and while it was designed primarily for use on thin sheet metals, it is also adapted for use on 0.5-in. diameter tension test specimens. The prick punches of this marker are of hardened steel, spaced 2 in. apart (±0.002 in.). Their points are ground to a total angle of 50 deg. so that the gage marks may be applied by merely striking the instrument with the palm of the hand and so that the marks will have a minimum effect on the mechanical properties of the specimen.

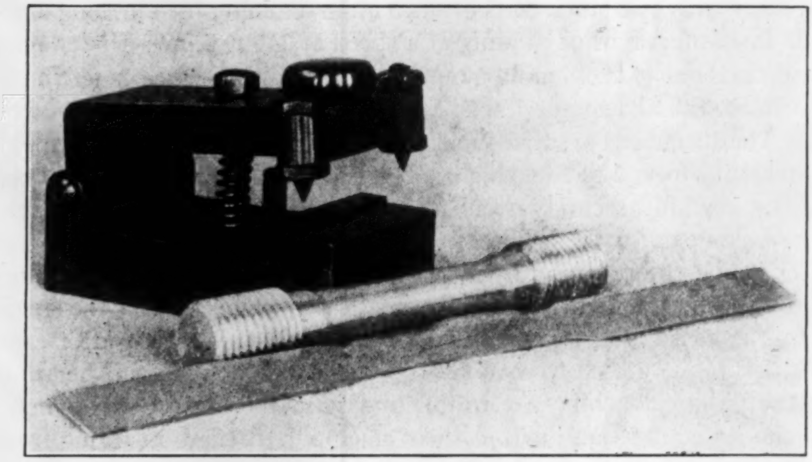

FIG. 4.—2-in. Gage Marker.

The placing of gage marks on specimens prepared from very thin metals (0.005 to 0.020 in.) involves considerable care to prevent the specimens being damaged sufficiently as to cause fracture through the gage marks. That is, the gage marks in such instances must be very shallow. With such gage marks difficulty is sometimes experienced in finding the marks after the specimen has been tested. In such cases pencil, ink or crayon marks may be drawn around the gage marks so that they can be positively identified after the specimen is tested. Usually two sets of gage marks are placed on a specimen. These may or may not be placed on opposite sides of the specimen but are both placed on the center line of the specimen and staggered approximately 0.25 in. Such procedure often eliminates the necessity for discarding specimens where the fracture might otherwise be outside of the gage length.

All thin sheet metal tension test specimens which we test are measured, using suitable micrometer calipers; that is, in no case do we use nominal dimensions. A definite attempt is made to keep all dimensions of the reduced section within plus or minus one per cent. This is comparatively easy to do in the case of the measurement of width, but for thickness it becomes rather difficult in routine commercial tests of material 0.015 in. or less in thickness. When the test specimens are properly machined, the dimensions at the middle of the reduced section are those used in determining the cross-sectional areas of the specimens. Additional measurements of both width and thickness are made at the ends of the reduced sections as a check on the machining operation.

Generally, we prepare three tension test specimens from each sample submitted, but in most cases only two of these are tested, the third being reserved for test in case of appreciable differences in the results obtained from the tests of the first two specimens.

TEST PROCEDURE

Testing Machines.—The tension specimens are tested in machines of either the hydraulic-pendulum-dynamometer or screw-power-lever type. Our preference is for the first type mentioned. All of the machines must meet the A.S.T.M. requirements for accuracy and sensitivity.[1] Machines having two or more capacity ranges are quite to be preferred for tension tests of thin sheet metals. Our experience has indicated that in the case of machines of two capacity ranges, the smaller range should not exceed 2000 lb. and the larger range should not be more than ten times the smaller. More satisfactory results are obtained with machines having four or more capacity ranges. We are using some Amsler machines, each having capacity ranges of 200, 400, 1000, 2000, 4000, 10,000 and 20,000 lb., which permit of testing very wide ranges of kind, grade and temper of thin sheet metals.

Gripping the Specimen.—Satisfactory tension tests of thin sheet metals can be obtained only when proper attention is given to the method of gripping the ends of the specimens. That is, the load must be applied uniformly and axially to the specimens. Generally speaking, none of the available commercial testing machines have suitable gripping facilities for making satisfactory tension tests of thin sheet metals in the thickness range previously indicated. This fact has been appreciated by others, notably, Anderson, Knerr and Otey in this country, and various designs of such grips or testing machine attachments have been devised to solve the problem. We designed and built our first set of special grips for thin sheet metals in January, 1920. Since then the original design has been improved upon appreciably and our present style of grips is shown in Fig. 5. These grips have a total capacity of 8000 lb. and handle the entire range of thickness of sheet metals under discussion.

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[1] Standard Methods of Verification of Testing Machines (E 4 – 27), 1927 Book of A.S.T.M. Standards, Part I, p. 793; also Part II, p. 921.

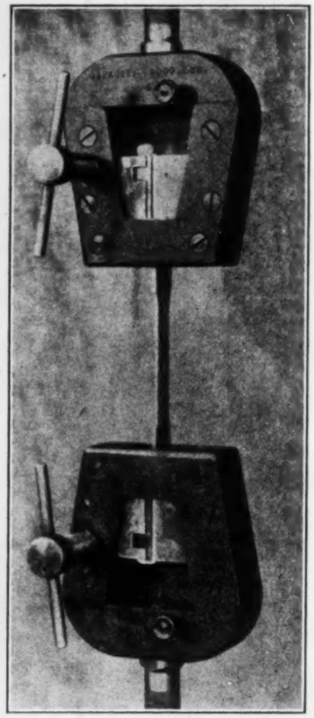

FIG. 5.—Special Grips for Thin Sheet Metals.

An important feature in any machine grips for thin sheet metals is the size and shape of the grip teeth. We have found that machined double-cut teeth of about 0.030 to 0.035-in. pitch are most satisfactory. These are appreciably finer than those usually provided. Such teeth hold the thicker specimens as well or better than coarser teeth and very much facilitate the removal of broken test specimens from the grips.

In making tests of thin sheet metals, more than ordinary precautions should be taken to see that the testing machine is sensitive and accurately balanced; that the grip teeth are clean; that the working parts of the grips are properly lubricated; and that the specimens are properly inserted in the grips before testing.

Speed of Testing.—As the rate of application of load often affects appreciably the tensile properties, especially the yield point, of metals, we have found it necessary to define a maximum value for speed of testing. This maximum value is 0.4 in. per minute for the pulling head of the testing machine when using the specimen shown in Fig. 1, and the speed generally used is approximately half this value. When using testing machines requiring manual balancing of the load, still slower speeds are advisable for best results.

The need for the requirements and precautions just discussed can hardly be over emphasized. This becomes apparent when it is considered that many of the tension specimens with which we are concerned require total loads of less than 200 lb. and often less than 100 lb.

Determination of Yield Point.—The tensile properties we usually determine for thin sheet metals include tensile strength, elongation in 2 in., yield point, proportional limit and Young's modulus of elasticity. Experience has shown that the reduction of area is so difficult of determination in the case of thin sheet metals as to be of little or no value. Of the properties determined, the so-called "yield point" presents the most difficulties, chiefly because of disagreement as to its definition and the absence of a satisfactory and rational method for its determination with respect to different metals. This is especially true of metals that have no well-defined hook or arrest in their stress-strain diagrams as is normally found for hot-rolled low-carbon steel. Although, admitting the inaptness of "yield point" for designating a tensile property of metals other than the exception just noted, yet for lack of better this term will be used in the discussion to follow. It may be pointed out, however, that the first method discussed, for determining this property, might be considered suitable in many instances for defining a satisfactory elastic limit.

The method we have developed for determining the yield point of metals that show a gradual departure in their stress-strain diagrams from the initial slope or modulus line, involves the use of a factor dependent upon the average initial or Young's modulus of elasticity of the material. This factor is obtained from the following empirical equation:

$$f = \frac{0.15\,E}{10^9}, \text{ or}$$

$$f \text{ (in per cent)} = \frac{0.15\,E}{10^7} \qquad \text{(1)}$$

where f = the yield point factor and E = Young's modulus for any given metal.

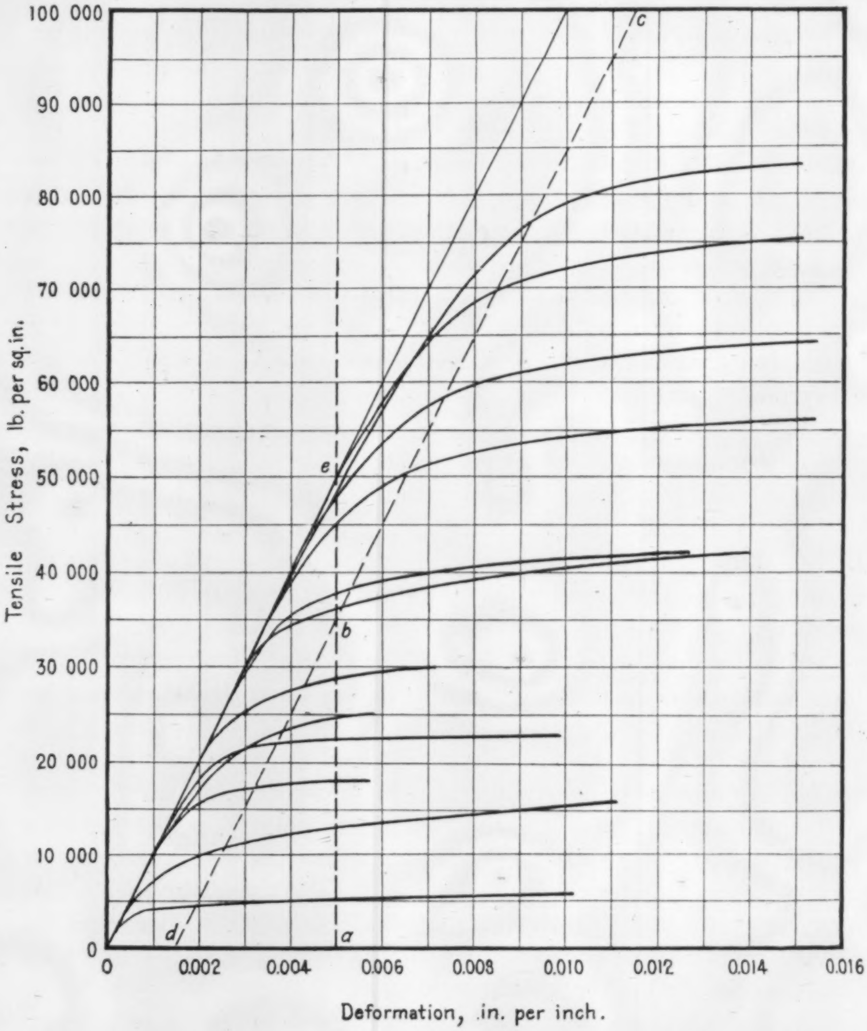

FIG. 6.—Tensile Stress-Strain Curves of Wrought Aluminum and Aluminum Alloys.

The stress corresponding to the point on the stress-strain diagram where the curve has departed from the initial modulus line produced, by an amount equal to the factor for that metal, is taken as the yield point of the metal. This is, perhaps, better understood by reference to Fig. 6, which shows a number of stress-strain curves for various tempers and kinds of aluminum alloys, all in the wrought condition. An average value for Young's modulus for aluminum and its alloys is 10,000,000 lb. per sq. in. Substitution of this value in Eq. 1 gives a factor of 0.15 per cent, corresponding to a unit deformation of 0.0015 in. per inch. Drawing the line *dc* 0.0015 in. from and parallel to the initial modulus line, gives the locus of all yield point values for aluminum according to our definition. The actual values may be read opposite the intersections of this line with the different stress-strain curves.

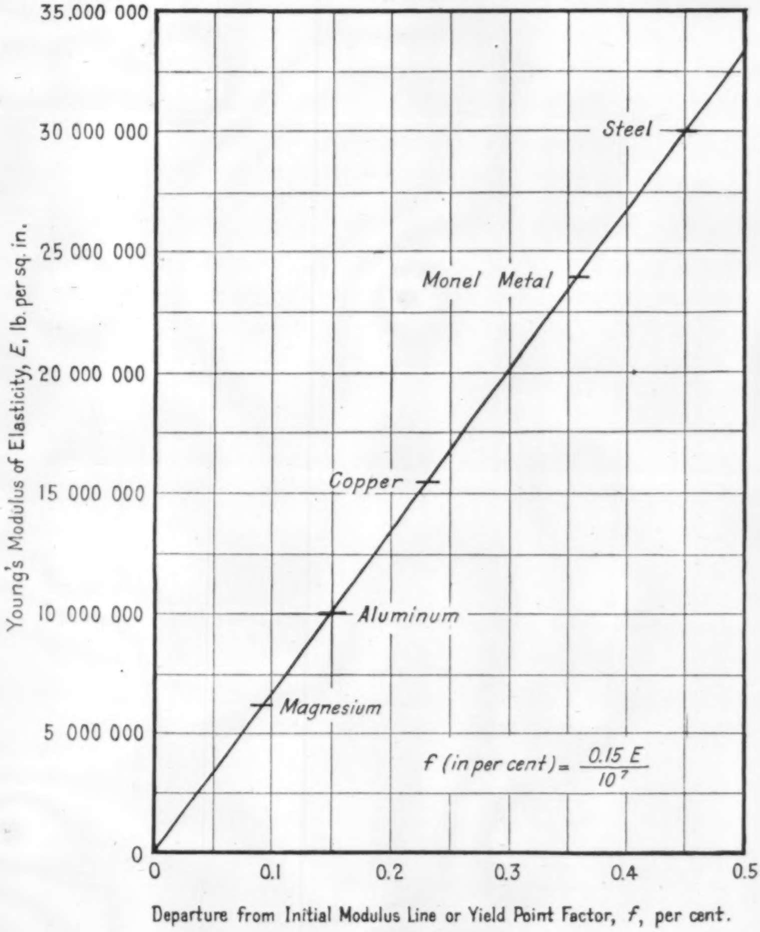

FIG. 7.—Young's Modulus - Yield-Point Factor Curve.

The graph of Eq. 1 is shown in Fig. 7, and suggested values of the yield-point factor are indicated on it for a number of different metals. Using these factor values, yield points of the same metals have been located on the stress-strain curves shown in Fig. 8, all curves being plotted to the same scales.

While this method of determining the yield point is not proposed as a final solution of the problem, yet it would appear more rational and comprehensive than others previously offered. We have used it for the past six or seven years with quite satisfactory results. In routine testing, we have found that it can be readily applied by first determining from a representative few stress-strain tests of a given kind, temper and condition of a metal, the average unit deformation under load corresponding to the average yield point of the material.

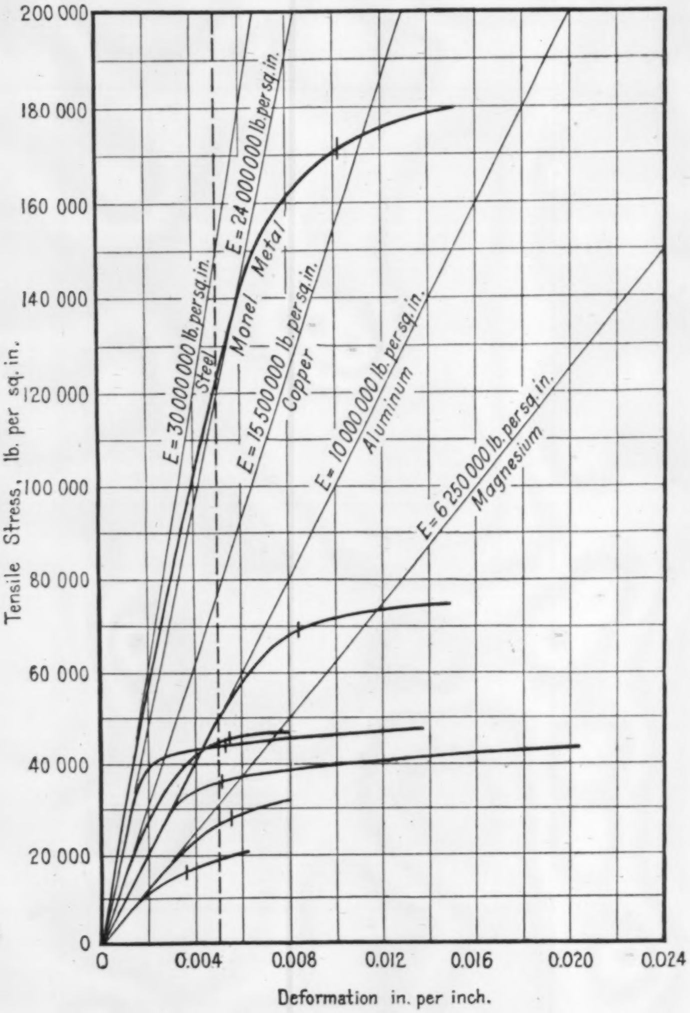

FIG. 8.—Tensile Stress-Strain Curves of Various Metals.

Sharp-pointed dividers are then set to a distance equal to this deformation value multiplied by the gage length, plus the gage length used. One point of the dividers is then placed in one of the prick punch marks of the gage length and the stress in the specimen determined when the other point fits into the mark at the other end of the gage length. This stress is taken as the yield point of the material. When the gage length used is short or the material being tested has a low yield point, or both, a suitable extensometer is used instead of the dividers for determining the required deformation. In most cases we have found that a minimum value for the deformation under load of 0.5 per cent is satisfactory for routine inspection tests of non-ferrous metals. The locus of the yield point values for aluminum and its alloys then becomes *abc* in Fig. 6.

In addition to the method just described for determining the yield point, we use a number of other methods that have been correlated with one another, using the first method as a basis.

An extensometer of the indicating dial type is attached to the specimen and load applied at a uniform rate. When the hand of the extensometer first shows a marked increase in its rate of movement, the load on the specimen is recorded as the yield point in pounds. The unit value is obtained in the customary manner. When suitable attention is given to testing speeds, kind and temper of metal, and with some experience in its use, this method will give satisfactory results.

When the testing machine is provided with a suitable autographic attachment, the yield point can be obtained from the load-deformation curves by selecting a point on them corresponding to the value which would be obtained if the first method described were used. This procedure involves an appreciable personal equation, so again, experience is necessary for satisfactory results.

The yield point values of certain tempers or conditions of most non-ferrous metals often can be determined satisfactorily by using a suitable extensometer and noting the load on the specimen when the deformation is 0.5 per cent. This method is not satisfactory, however, for yield point values in excess of about 20,000 lb. per sq. in. for magnesium, 35,000 lb. per sq. in. for aluminum, and 65,000 lb. per sq. in. for copper or brass.

The yield point of some tempers of low-modulus non-ferrous metals can be approximately determined by the use of dividers and a watch. The dividers are set 2 in. apart, one point inserted in one of the gage marks of the specimen, then short scratches made on the specimen with the other point at equal intervals of time, usually five or ten seconds, during the testing of the specimen. When a perceptible increase in the distance between scratches is observed the load on the specimen is noted and recorded as the yield point.

For certain metals of specific temper, an approximate yield point may be determined by noting the first point where there is a perceptible decrease in the rate of taking load by the specimen. This method can be used to best advantage when the testing machine is provided with a dial-type load-indicating mechanism.

Determination of Modulus and Proportional Limit.—The determination of Young's modulus and the proportional limit of thin sheet metals involves the use of suitable extensometers as in the case of other forms of metals. Extensometers of the Martens' mirror type can be used in tension tests of thin sheet metals quite readily, but comparatively few laboratories are equipped with such instruments. Furthermore, the use of this type of extensometer does not permit of rapid routine testing. Extensometers of the indicating-dial type are better suited for routine tests, especially where the yield point only is desired and not the modulus value. Dial type extensometers, however, are usually arranged for attachment to the specimen by means of conical shaped points. With this arrangement these extensometers cannot be satisfactorily attached to the thinner sheet metals if the points are applied so as to grip the thickness of the specimens. Attachment to the edges of the specimen entails some difficulties that usually can be overcome if special blocks, similar to those shown in Fig. 9, are used on each point of the extensometer. A set of these attachment

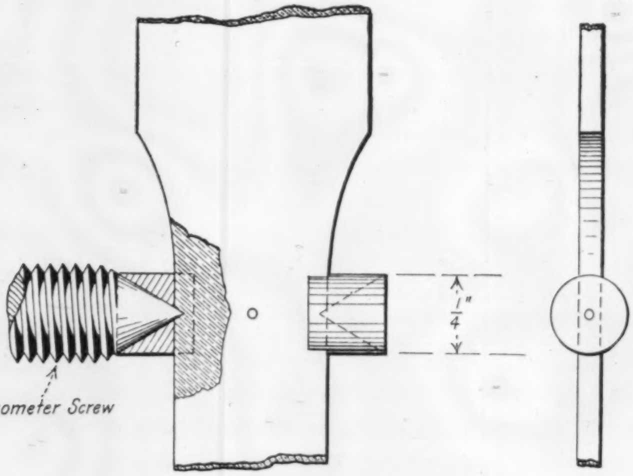

FIG. 9.—Extensometer Attachment Blocks for Sheet Metals from 0.008 to 0.064 in. Thick.

blocks made for sheet metal of the following thicknesses has been found satisfactory for all intermediate thicknesses:

B. & S. GAGE NUMBER	THICKNESS, IN.
14	0.064
16	0.051
18	0.040
20	0.032
22	0.025
24	0.020
26	0.016
28	0.013
30	0.010
32	0.008

If satisfactory values for modulus and proportional limit are expected, the extensometers used in their determination must give deformations within the required limits of error. In order to determine the errors and maintain the required accuracy, the extensometers should be calibrated frequently. Unfortunately, suitable extensometer calibrating devices are not available in the open market. We have, therefore, designed and built an instrument for this purpose and have found it quite satisfactory. Total deformations to the nearest 0.000002 in. are determined with it and a total range of 0.04 in. is provided. This instrument will be described in detail elsewhere.

Determination of Elongation.—After breaking the specimens, measurements of elongation are made. We have found that this can be done most readily by using sharp-pointed dividers and a scale graduated in fiftieths of an inch. With such a scale hundredths of an inch can be estimated more quickly and accurately than if the scale were actually divided into hundredths of an inch. A scale of the draftsman's type wherein the zero or first division is some distance from the end of the scale, is to be preferred because it permits of setting one point of the dividers directly on the zero mark, thus further helping to eliminate errors in reading elongation values.

In addition to elongation measurements, record is made concerning type and location of fracture and presence and character of defects, if any. These notations, together with the original dimensions of test specimens, the maximum total loads in pounds, and usually the yield points in pounds, comprise the record of test observations on the data sheets. From these data the mechanical properties are calculated using a slide rule or calculating machine that will give at least four significant figures in the unit values for tensile strength and yield point. The elongation is usually expressed to the nearest 0.5 per cent. In these calculations, however, a definite attempt is made to see that they are not carried out appreciably beyond the point warranted by testing errors. Checking of test data is done by a second person, both as regards calculations and omission of information or observations. Mistakes in the recorded data may be crossed out but not erased. A good test of this requirement is to consider whether or not the data record will prove to be acceptable from a legal view point. Dates are considered essential on all test data records.

Stress-strain data from tests of a given kind of metal preferably should be plotted to similar or identical scales if satisfactory comparisons of results are to be obtained. This has been appreciated by many others but our experience has shown the need for further emphasis, particularly when dealing with metals having no well-defined yield points.

USES OF RESULTS

Using the testing procedure and equipment just described, it has been possible to materially increase the number of tests per man-hour, to increase the efficiency of the testing machines and to decrease both the number and magnitude of errors in the test data in making tension tests of thin sheet metals. Under such conditions, it has been possible to extend the scope and add to the usefulness of the tension test. We have found the tension test valuable in the inspection of thin sheet products, where it is used as a check on the temper, condition and uniformity of the material. Comparison of these data from different sheet mills often serves as a check on similar products made under different fabricating conditions. In many instances, the tensile properties are used as criteria in studying and making changes in the fabrication practice of an existing product or in the development of a new product.

Given a large amount of accurate tensile property data, obtained from routine tests extending over a considerable period of time, the task of determining suitable specification and engineering design values from such data is materially lightened. A definite knowledge of the tensile properties of a given kind and temper of sheet metal also is often of considerable assistance in comparing its drawing, forming and working properties with other kinds or tempers of sheet metals of known tensile properties.

The examples of usefulness of the tension test thus briefly mentioned are but the more important ones which experience has so far demonstrated. With a more general application of the test to thin sheet metals, it is believed that many additional benefits will accrue to both producer and consumer of sheet metal products.

DISCUSSION

Mr. H. F. Moore.[1]—At this meeting there have been presented several papers dealing with the development of a tension specimen for thin sheet metals. I wish to point out how much work has been involved in this apparently simple problem. Thousands of tests have been made, with the necessity for reducing and tabulating the accompanying test data. Whether the specimen proposed as standard proves to be the final form of specimen adopted or not, I believe that the careful experimental study followed in studying the thin sheet specimen is a distinct advance over the somewhat haphazard method followed in developing many of the specimens now in use. The men who have been studying the testing of thin sheet metal have set a good example for testing engineers who have to develop new test specimens. **Mr. Moore.**

Mr. J. R. Townsend[2] (*presented in written form*).—The writer had the privilege some time ago of visiting Mr. Templin's laboratory and seeing in operation the methods described in this paper. These methods have accomplished much in reducing the cost of the tension test on thin sheet metals and have made possible a wider use of the tension test for inspection of such materials. **Mr. Townsend.**

Similar methods to those given in this paper have been placed in use by the Bell Telephone Laboratories. Inasmuch as the amount of routine tension testing required does not justify the use of a special milling machine for the preparation of cross-milled specimens, we have adopted a modification of this scheme that may be installed in any machine shop at small cost. It is felt that this modified method may be used to advantage by many testing laboratories.

The specimens are sheared, sawed or blanked from sheet stock in the form of rectangular strips $\frac{11}{16}$ in. wide and 8 in. long. The specimens are then clamped together to make a pack about $1\frac{1}{2}$ in. thick and are drilled at each end with a 0.181-in. drill, the drilling being made on the center line $\frac{1}{4}$ in. from the end of the specimens. The specimens are then clamped between two bars which have a profile as shown in the accompanying Fig. 2, designed to support the specimens but clearing the profile cutter, the pins which join the two bars passing through the holes in the specimens. The horizontal pin bearing against one end of the clamp is for the purpose of locating the clamp longitudinally when it is turned over so that the cut on the second side of the specimen is exactly opposite the cut on the first side. This assembly is then clamped between the vise of a milling

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[1] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] General Development Laboratory, Bell Telephone Laboratories, Inc., New York City.

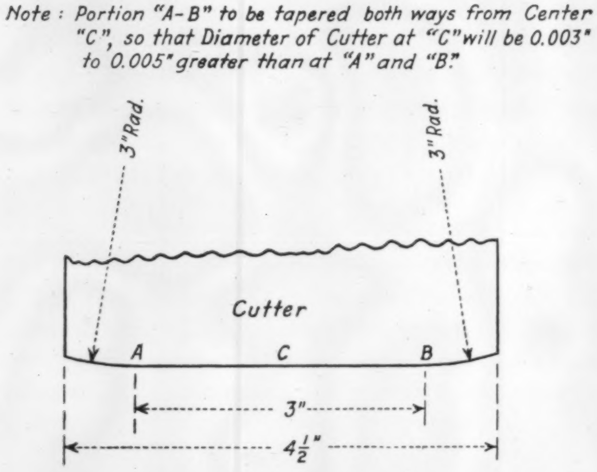
Note : Portion "A-B" to be tapered both ways from Center "C", so that Diameter of Cutter at "C" will be 0.003" to 0.005" greater than at "A" and "B."
3" Rad.
3" Rad.
Cutter
A
C
B
3"
$4\frac{1}{2}$"

FIG. 1.—Contour of Forming Cutter for Milling Tension Test Specimens.

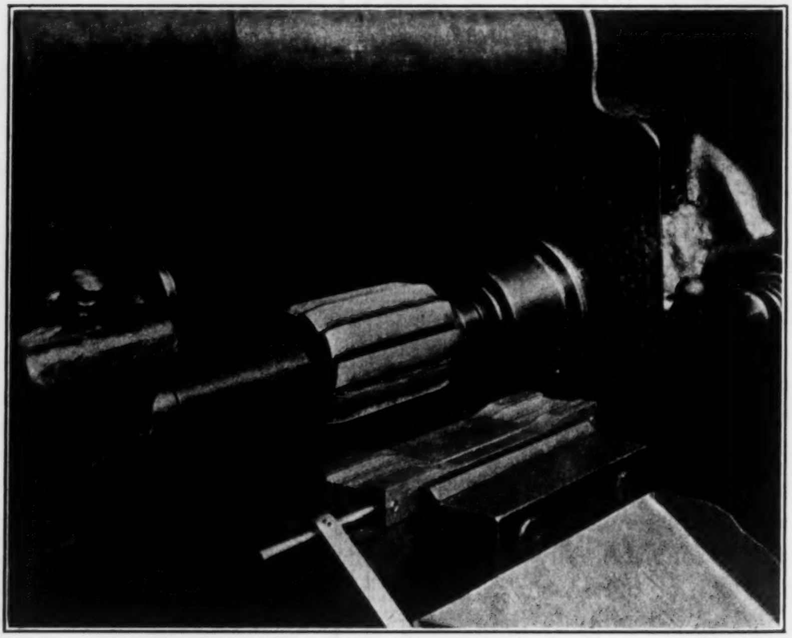

FIG. 2.—Assembly for Milling Tension Test Specimens.

Mr. Townsend.

machine as shown in the accompanying Fig. 2 and is located by the pin indicated. The gage lengths and fillets are cut by a profile milling cutter shown in the accompanying Fig. 1. The form of milling cutter employed is different from that indicated in Mr. Templin's paper. It has been found that a straight milling cutter is entirely satisfactory and has much to recommend it by reason of the fact that it is easy to regrind or resharpen such a cutter with simple grinding equipment. The method of milling the specimens consists in taking one cut with the set-up as shown in Fig. 2, followed by a finishing cut designed to compensate for the elasticity of the machine, to remove any chatter marks and give a smooth surface. The clamped specimens are then turned over and are finished in the same manner on the other side.

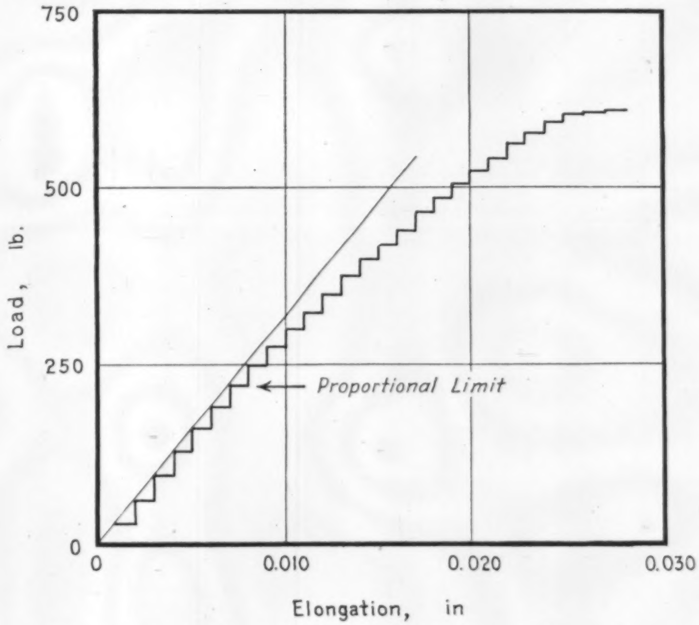

FIG. 3.—Load-Elongation Curve for Half-Hard Sheet Brass.

It is believed that the method of determining the yield point as described in the paper has much to recommend it, but the application of such a method requires considerable interpretation in design work. What the designer is particularly interested in is elastic range, or the extent to which the material may be loaded without suffering deformation. It is believed that the proportional limit method of measuring the extent of the elastic range gives as good an indication as can be obtained by a short time test. The determination of the proportional limit is rather an expensive procedure if carried on in

Mr. Townsend. the usual manner, since one operator is required to operate the machine and another to observe and record the extensometer readings.

The method we have worked out requires but one operator to carry on the test and the results are as accurate as those that can be determined with the best extensometers now in use. In this method the load is indicated by axial movement of a cylinder or by axial movement of a pen along the surface of paper attached to the cylinder. In either case, rotation of the cylinder corresponding to elongation is determined by a ratchet wheel and an electromagnetic stepping device, the latter producing unit rotation of the cylinder for each time a key is pressed by the operator. The operator observes the reading of the extensometer and each time the extensometer reaches a previously determined unit elongation he presses the key. The resulting curve is a series of steps. A straight line drawn through the points indicated on the left-hand side of the series of steps as shown in the accompanying Fig. 3 will pass through all points where the unit load is equal to unit elongation. Where the line fails to pass through these points, the proportional limit is indicated. Since the unit rotation of the cylinder is dependent upon the fineness of the

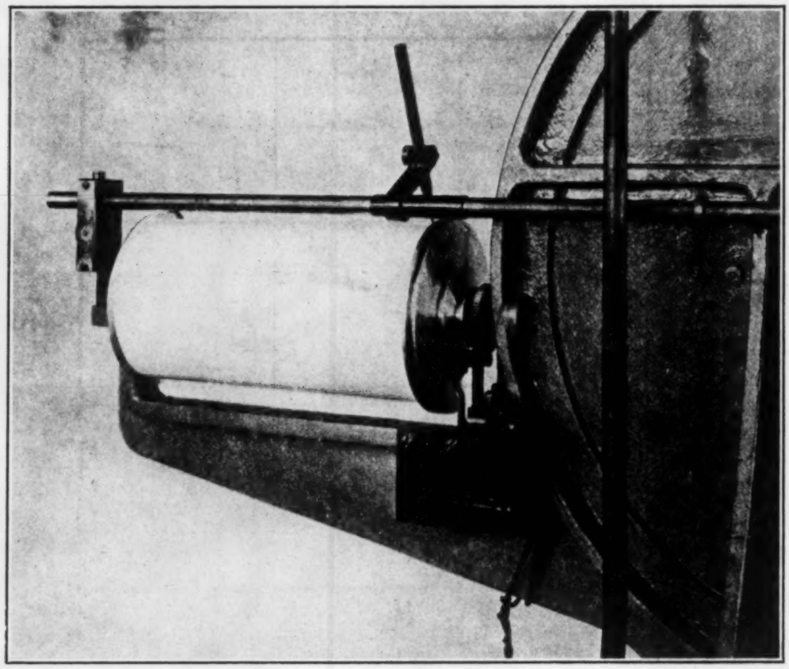

FIG. 4.—Application of Extensometer to Amsler 20 000-lb. Universal Testing Machine.

reading that can be made on the extensometer and the number of teeth on the ratchet wheel, it is obvious that any practical degree of refinement of reading may be provided.

Mr. Townsend.

A specific application of this method to an Amsler 20,000-lb. universal testing machine is shown in Fig. 4. Figure 5 shows the machine in operation. This same form of device may be applied to the beam type of testing machine by placing the marker on the poise and rotating the cylinder as before.

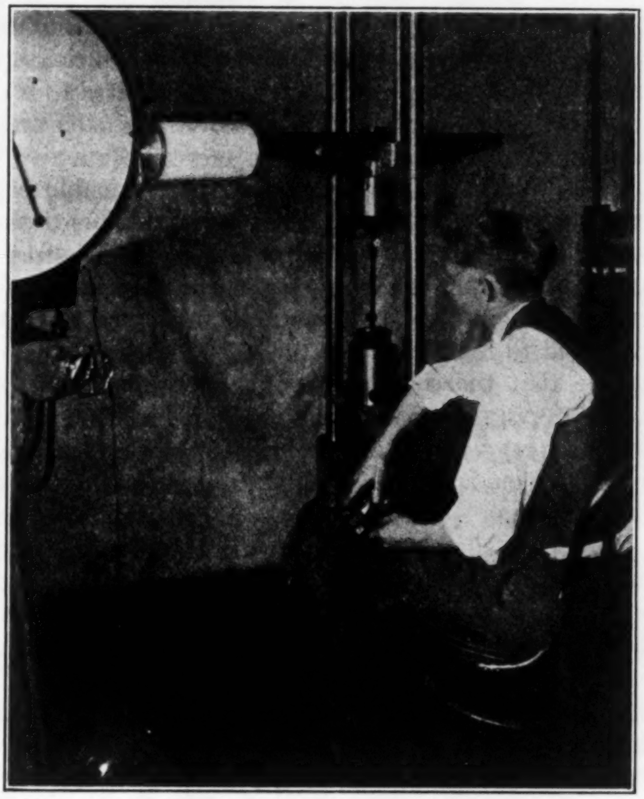

FIG. 5.—Showing Method of Operating Testing Machine.

Another consideration which is of very great importance in tension testing of sheet metals is the question of percentage accuracy with particular reference to methods of measuring the width and thickness of the specimen. It is fairly easy to measure the width of a tension test specimen to an accuracy of 1 per cent since this involves reading the ½-in. dimension to 0.005 in. To measure the thickness of a sheet metal 0.005 in. thick to 1 per cent accuracy requires that this thickness be measured to 0.00005 in., and it is obvious that

Mr. Townsend.

the ordinary micrometer is of little value. A measurement to have an accuracy of 0.00005 in. must be measured with an instrument sensitive to 0.000005 in. Such a fine measurement is impractical to make in view of the equipment involved and other considerations. It is considered practical, however, to measure to ± 0.00005 in., that is to the nearest 0.0001 in., which in turn will require an instrument sensitive to 0.00001 in. It is obvious from this discussion that it is essential that the measuring equipment shall correspond to the degree of accuracy that is desired. Not only must the measuring equipment correspond, but the technique must be carefully stated; for example, the pressure between the measuring mandrels and the area of the mandrels is very important.

Mr. Whittemore.

MR. H. L. WHITTEMORE[1] (*by letter*).—This valuable paper is, I believe, unique in that it presents the results of years of work in a successful attempt to put the routine testing of a specialized product on a quantity production basis without sacrificing accuracy of results. In most testing laboratories, both research and commercial, too little attention is paid to reducing testing costs. Materials engineers, too often, assume that the cost of carrying on the work is fixed, like taxes, and is a necessary expense. Efforts to reduce the cost of testing work are well worth while because only in that way can the control of the product be made so effective that unsatisfactory material does not get into service.

Mr. Templin's efforts have, I understand, resulted in the development of methods which give almost identical values when specimens from the same sheet are tested by different men, using different machines in different laboratories.

One should distinguish between methods which affect the test results and those which simply reduce the cost. For example, the use of profile milling cutters is hardly justified if only a few specimens are machined at infrequent intervals. We have found that the cost of machining some of our standard specimens is about ten times the cost of testing them. It seems probable that by using the methods described in this paper, the cost of machining specimens from thin sheet metal may be as low as one-tenth the cost of testing them. This makes it practicable to obtain many more data and increase the value of the investigation.

Using the marker shown in Fig. 4 of the paper, the distance between the gage marks will be within very close tolerances, certainly much closer than if the usual marker is used having points which slide in the frame, each being struck, in succession, by a hammer to indent

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[1] Chief, Engineering Mechanics Section, VI-5, U. S. Bureau of Standards, Washington, D. C.

Mr. Whittemore.

the specimen. Although the construction of this improved marker offers no difficulties it would be much more widely used if it were a commercial commodity.

It is interesting to note the preference for the hydraulic pendulum-dynamometer type of testing machine having several capacity ranges for testing sheet metals because, as a usual thing, engineering apparatus built in a foreign country is not extensively used in this country. The European builders of testing machines have developed testing machines which differ radically from ours. Probably few materials engineers in America have used the hydraulic-pendulum-dynamometer type of machine sufficiently to form an opinion of its advantages. It is unfortunate that the advantages and disadvantages of each type of machine have not been more carefully studied and are not better understood in this country.

The outstanding contributions of Mr. A. H. Emery in this field are widely recognized and we cannot afford to fall behind in this important branch of engineering technology. We must not forget, however, that conditions in Europe make it possible to make apparatus in small quantities which cannot be made in this country at a reasonable price unless the number which can be sold is very greatly increased so that modern methods of quantity production are justified.

The need for grips especially designed for thin sheet metal specimens is shown by the fact that Templin, Knerr, and Otey each found it impracticable by using commercial grips to obtain the accuracy which they considered necessary.

The formula for determining the yield point given in this paper should receive careful consideration by other materials engineers. Some method is greatly needed which will give, for different observers using different equipment, closely comparable results. The determination of the stress at which the elongation reaches a given value is thoroughly practicable, as inexpensive apparatus is on the market which can be used on specimens having a 2-in. gage length. Most of these instruments are sufficiently accurate and some have the advantage that they can instantly be attached or detached from the specimen without marking the gage points on the specimen.

If the A.S.T.M. specifications gave the elongation which should not be exceeded at a given (yield point) stress, one of these instruments could be used. The cost of making the test would be no greater than when the yield point is found from the "drop of the beam" of the testing machine, and a large personal element would be eliminated. Some satisfactory method of determining the yield point should be worked out by this Society and all specifications for metals should make its use mandatory.

Mr. Whittemore Mr. Templin has made very helpful suggestions which could be followed to advantage by Committee E-1 on Methods of Testing.

Mr. Marshall. MR. C. H. MARSHALL[1] (*by letter*).—Mr. Templin's paper presents a very interesting description of good testing practice as worked out in a laboratory, which does sheet metal testing in large quantities. The same methods, however, apply equally well where fewer samples are to be tested. Many of the details so frequently overlooked, but necessary to successful testing of thin sheet materials, are brought out in this paper.

One of these details is the matter of scratching identification numbers or marks on thin sheet metal test samples. Incorrect test results frequently are obtained due to incorrect gripping caused by identification marks stenciled on the ends.

The size of grip teeth also is quite important. Our experience has shown that when testing thin sheet materials (and wire also) the use of fine teeth is absolutely necessary if good results are to be obtained.

When testing samples of very thin materials, the use of self-aligning grips becomes quite important and those described are the best we have seen. They are simple in construction, easy to use and are so constructed that the grips can easily be removed for cleaning or replacement.

We have found the method of transverse milling by using a special profile cutter to be the most practical way of preparing tension test specimens. The extra expense of the form cutters is more than repaid by the greater ease of doing the work, even where relatively few samples are to be machined.

Mr. Templin's method for locating yield point is interesting and will be found less difficult to apply than one might think from a casual reading of it. This method gives a point which is considerably higher than the elastic limit as located by the "Johnson" method (or by other methods) and very near to or coincident with the "drop of the beam" if this is present. It should therefore give a satisfactory yield point value where there is no drop of the beam. An additional advantage of this method is the fact that the point located is at the intersection of a straight line with the curve. This is a definite point and will not vary with the scale to which the curve is plotted as does the proportional limit or end of the straight line, which is very difficult to locate and will appear higher if the curve is plotted to a small scale and lower if plotted to a larger scale.

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[1] **Testing Engineer, Chemical and Physical Testing Laboratory, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.**

TENSION TEST SPECIMENS FOR SHEET STEEL

BY J. T. NICHOLS,[1] E. S. TAYLERSON,[2] AND J. C. WHETZEL[3]

SYNOPSIS

An extensive series of tension tests has been carried out on sheet steel of various grades and thicknesses, making use of seven different test specimens, and also varying the radius of fillet and the end width. The results of the investigation show that the yield point and tensile strength are only slightly affected by the shape of the specimen. For material of the same grade, the elongation is a definite function of the size of the gage section. This relation for sheet steel is in agreement with the work of previous investigators on other metals.

The precision of the results for different designs of test specimen indicates that a gage section 2 in. long and $\frac{1}{2}$ in. wide is suitable for testing sheet steel.

INTRODUCTION

A recent survey[4] showed that there are in use, in this country, thirty-four different tension test specimens for testing thin sheet metal. These specimens vary in gage length from 2 to 8 in., and in gage width from $\frac{3}{16}$ to $1\frac{1}{2}$ in. It is impossible to compare test data based on this wide variety of specimens, unless one has data regarding the effect of size and shape of specimen on tensile properties.

The work of the Section on Testing Thin Sheet Metals of Committee E-1 on Methods of Testing of the American Society for Testing Materials leading to the adoption of a standard test specimen made it necessary to determine the suitability of the proposed design for the testing of sheet steel.

The investigation covered by this report was divided into two parts: The effect of variables external to the gage section, such as radius of fillet and end width of test specimen within the limits proposed by Committee E-1;[5] and the effect of variations within the gage section, such as gage length, gage width and thickness.

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[1] Physicist, Research Laboratory, American Sheet and Tin Plate Co., Pittsburgh, Pa.

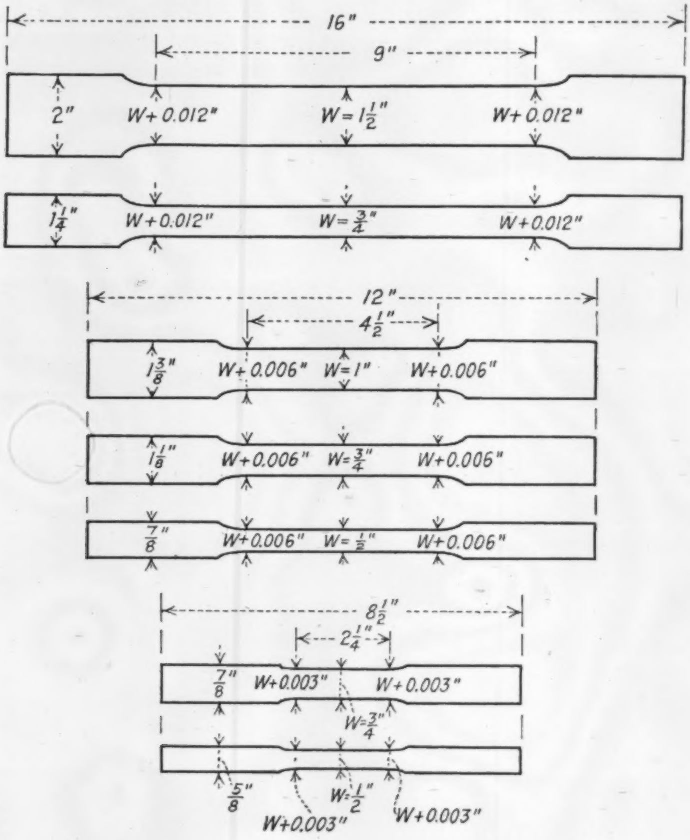
[2] Engineer of Tests, American Sheet and Tin Plate Co., Pittsburgh, Pa.

[3] Manager of the Research Laboratory, American Sheet and Tin Plate Co., Pittsburgh, Pa.

[4] Report on Testing of Thin Sheet Metals, Appendix II, Report of Committee E-1 on Methods of Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 531 (1926).

[5] *Ibid.*, p. 533 (1926).

The first part of the investigation confirmed the results of more extensive tests on other metals,[1] as the results showed that the effect of small changes of radius and end width could be neglected. These results include about 1200 tests covering four thicknesses of low-carbon sheet steel and seventeen end designs for the 2 by ½-in. gage section.


16"
9"
2"
W+ 0.012"
W = 1½"
W+ 0.012"
1¼"
W+0.012"
W = ¾"
W+0.012"
12"
4½"
1⅜"
W+ 0.006"
W = 1"
W+0.006"
1⅛"
W+0.006"
W = ¾"
W+0.006"
⅞"
W+0.006"
W = ½"
W+0.006"
8½"
2¼"
⅞"
W+0.003"
W+ 0.003"
W = ¾"
⅝"
W = ½"
W+0.003"
W+0.003"

All Radii are 1½"

FIG. 1.—Form of Test Specimens.

The second part of the investigation, namely, the determination of effect of size of gage section, was limited to the seven specimens illustrated in Fig. 1, which represent those more widely used.

Samples from which these specimens were machined were cut from a wide variety of commercial steel sheets, as well as from material taken out of the line of production during manufacture. Figure 2 is a diagram showing the locations of the specimens on the sheets, planned to minimize effects due to variation in material. Twenty transverse and twenty longitudinal specimens of each of the seven designs were obtained from each of the fifteen samples varying in grade or thickness: 4200 in all.

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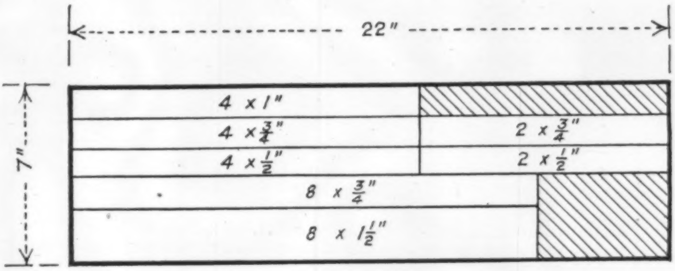
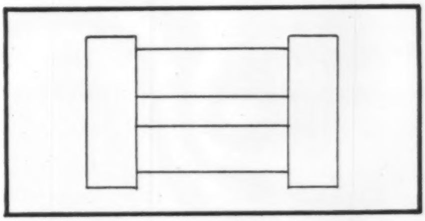
[1] R. L. Templin, "Effects of Size and Shape of Test Specimen on the Tensile Properties of Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 378 (1926).



Distribution of Specimen Blanks in Rectangle.



Distribution of Rectangles in Sheet.

FIG. 2.—Positions of Samples in Sheet.

Milling cutters with the profiles shown in Fig. 1 were used to machine the specimens over their whole length. Stresses were computed on the basis of actual measurements of thickness and width of gage section. Yield point, tensile strength and percentage of elongation in the various gage lengths were determined. An Amsler testing machine having seven different sensitivities and capacities ranging from 200 to 20,000 lb. was used for all tests. Autographic load-deformation diagrams were made for each specimen. The stress at which the specimen elongated without increase of load, as indicated on the load-deformation diagram, was arbitrarily selected as the yield point.

EFFECT OF SIZE AND SHAPE OF GAGE SECTION ON TENSILE PROPERTIES

In order to study the effect of varying gage length, width and thickness, the results of these tests have been plotted against the slenderness ratio, that is, the ratio of the gage length to the square root of the cross-sectional area of the specimen.

Yield Point and Tensile Strength.—As no appreciable variation in yield point or tensile strength was observed, data on these determinations are omitted, as they are in agreement with previous work on other metals.[1]

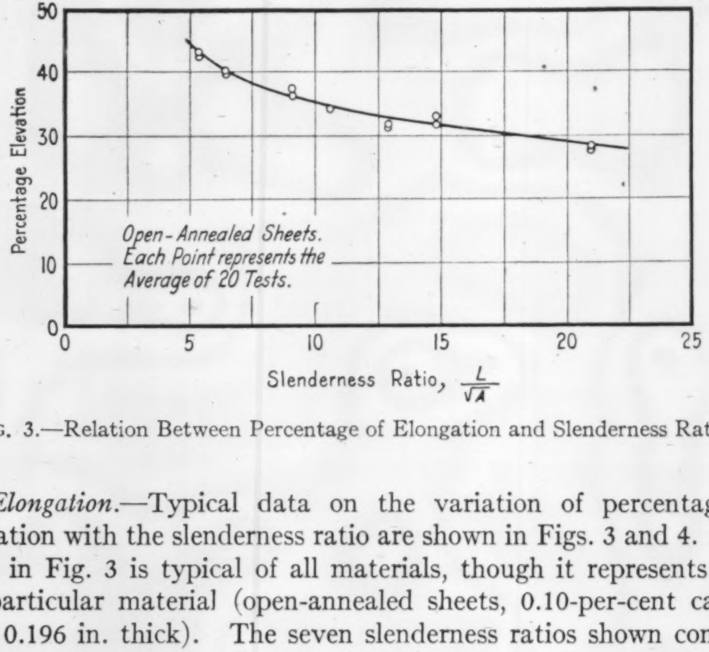


FIG. 3.—Relation Between Percentage of Elongation and Slenderness Ratio.

Elongation.—Typical data on the variation of percentage of elongation with the slenderness ratio are shown in Figs. 3 and 4. The curve in Fig. 3 is typical of all materials, though it represents only one particular material (open-annealed sheets, 0.10-per-cent carbon steel, 0.196 in. thick). The seven slenderness ratios shown conform to the seven specimens investigated. The two points indicated for each slenderness ratio show the values of percentage of elongation for longitudinal and transverse specimens, each point representing the average of twenty determinations.

In Fig. 4, each line represents one particular material of one particular thickness, selected from the fifteen materials tested, to illustrate variation in slope and intercept due to different grades and treatment of material. Each point represents the average of twenty determinations. The data for various materials shown in the log-

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[1] R. L. Templin, "Effects of Size and Shape of Test Specimen on the Tensile Properties of Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, pp. 382–385 (1926).

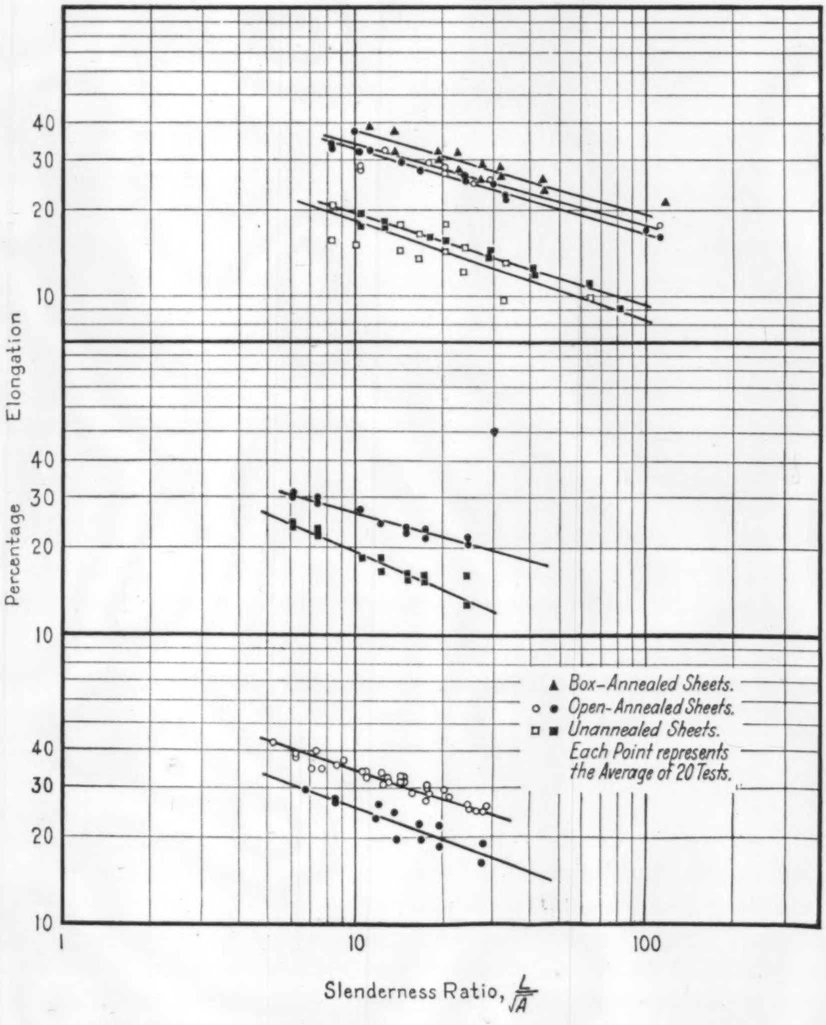

FIG. 4.—Relation Between Percentage of Elongation and Slenderness Ratio.

arithmic plots, Fig. 4, conform to Bertella's general equation for elongation:[1]

$$e = K\left(\frac{L}{\sqrt{A}}\right)^n = K\,S^n \qquad (1)$$

where e is the percentage of elongation, K and n are constants depending on the material, L the gage length, A the cross-sectional area and S the slenderness ratio $= \frac{L}{\sqrt{A}}$.

In order to obtain the ratio between elongations measured on specimens of the same material, but of different shape, it is only necessary to determine n because:

$$\frac{e_1}{e_2} = \left(\frac{S_1}{S_2}\right)^n \qquad (2)$$

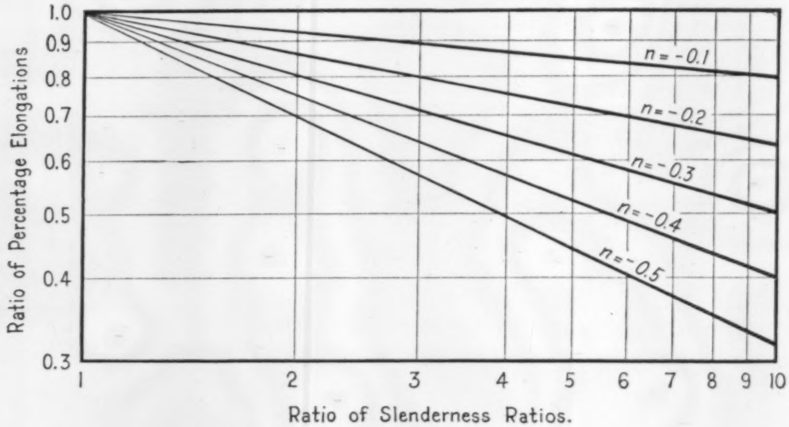

FIG. 5.—Chart for Solving Eq. 2.

Figure 5 is a chart for solving Eq. 2 by means of which the ratios of elongations can be found if the slenderness ratios and n are known. For example, n is found to be about -0.30 for commercial low-carbon annealed sheet steel. The slenderness ratio of the 8 by 1½-in. specimen is to that of the 2 by ½-in. specimen, of the same thickness, as 2.31 is to 1. From the chart we find that, for n equals -0.30, this ratio of slenderness ratios gives a ratio of elongations of 0.78; that is, the percentage elongation measured on an 8 by 1½-in. specimen of this material will be 78 per cent of that measured on a 2 by ½-in. specimen of the same thickness.

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[1] R. L. Templin, "Effects of Size and Shape of Test Specimen on the Tensile Properties of Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 390 (1926).

This chart is also useful for determining the relation between the percentages of elongation which would be expected from different thicknesses of materials of identical mechanical properties. The relationship of the elongations of annealed low-carbon sheet steel, using the seven specimens with which this investigation is concerned, is shown in Table I.

TABLE I.—RELATION OF PERCENTAGES OF ELONGATION OF VARIOUS GAGE SECTIONS OF SAME THICKNESS OF COMMERCIAL ANNEALED LOW-CARBON SHEET STEEL.

Gage Sections for Which Elongation Is Known	Multiplier for Calculating Percentages of Elongation of Various Sections ($n = -0.30$)						
	2 by $\frac{3}{4}$ in.	2 by $\frac{1}{2}$ in.	4 by 1 in.	4 by $\frac{3}{4}$ in.	4 by $\frac{1}{2}$ in.	8 by $1\frac{1}{2}$ in.	8 by $\frac{3}{4}$ in.
2 by $\frac{3}{4}$ in.	1.00	0.94	0.85	0.82	0.76	0.74	0.66
2 by $\frac{1}{2}$ in.	1.06	1.00	0.90	0.87	0.81	0.78	0.70
4 by 1 in.	1.18	1.11	1.00	0.97	0.90	0.87	0.78
4 by $\frac{3}{4}$ in.	1.22	1.15	1.03	1.00	0.93	0.90	0.80
4 by $\frac{1}{2}$ in.	1.31	1.23	1.11	1.07	1.00	0.96	0.86
8 by $1\frac{1}{2}$ in.	1.36	1.28	1.15	1.12	1.04	1.00	0.90
8 by $\frac{3}{4}$ in.	1.51	1.43	1.29	1.24	1.17	1.11	1.00

Example.—Required the percentage of elongation in 4 by $\frac{3}{4}$ in. when 25 per cent is given in 8 by $1\frac{1}{2}$ in. Find multiplier opposite 8 by $1\frac{1}{2}$ in. and under 4 by $\frac{3}{4}$ in., which is 1.12; 25 per cent x 1.12 = 28 per cent elongation in 4 by $\frac{3}{4}$ in.

TABLE II.—AVERAGE DEVIATIONS OF TENSION TEST RESULTS.

Each value represents average results from about 600 tests, both longitudinal and transverse, on fifteen kinds of annealed and unannealed sheet steel.

Specimen		Ratio, $\frac{\text{Gage Length}}{\text{Gage Width}}$	Average Deviation, ±, per cent		
Gage Length, in.	Gage Width, in.		Elongation	Yield Point	Tensile Strength
2	$\frac{3}{4}$	2.67	7.2	4.0	2.0
2	$\frac{1}{2}$	4.00	5.8	4.3	2.1
4	1	4.00	6.3	3.7	1.9
4	$\frac{3}{4}$	5.33	6.7	3.5	1.8
4	$\frac{1}{2}$	8.00	7.2	3.5	2.0
8	$1\frac{1}{2}$	5.33	6.3	3.5	1.9
8	$\frac{3}{4}$	10.67	9.4	3.5	1.9

EFFECT OF SIZE AND SHAPE OF GAGE SECTION ON PRECISION OF TENSION TEST RESULTS

As previously stated, each percentage of elongation, yield point and tensile strength reported in this investigation is the average of twenty determinations. The precision with which these quantities were determined was calculated and the average deviations averaged for tests made with each size and shape of specimen of all materials. These data are given in Table II and are plotted in Fig. 6.

From Fig. 6(*a*) it will be seen that the precision with which tensile strength and yield point are determined is only slightly affected by the gage length if this is equal to or greater than 2 in. Gage lengths of 1 in. and shorter would probably give unsatisfactory precision.

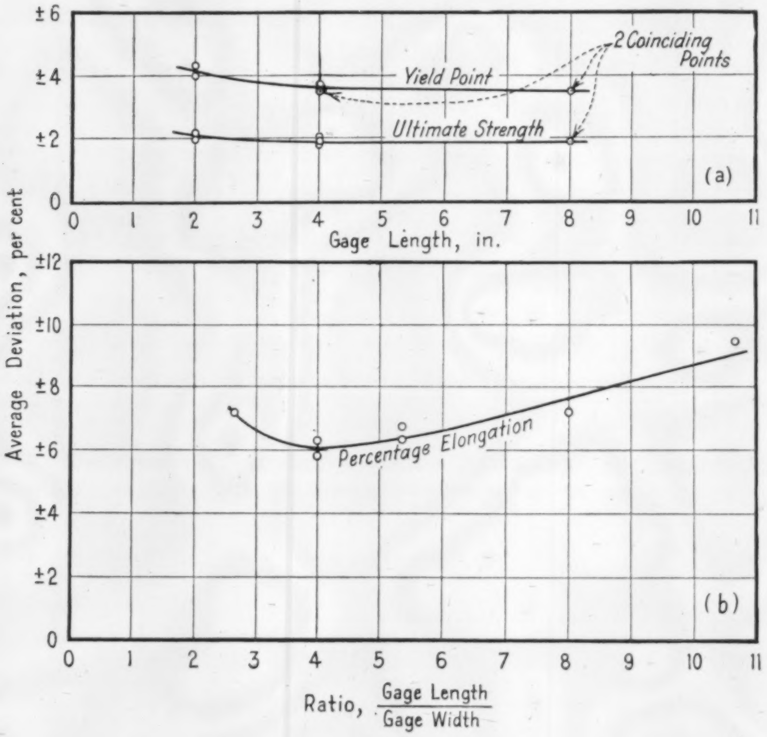

FIG. 6.—Effect of Size and Shape of Gage Section on Precision of Results.
Each point represents the average of 600 tests on fifteen materials.

The precision with which the percentage of elongation is measured is apparently a function of the ratio of gage length to gage width. It would be expected that short, wide specimens would give unreliable elongations and it is interesting to see from Fig. 6(*b*) that long, narrow ones are also less precise. The maximum precision, or minimum average deviation, is seen to occur at a ratio of gage length to gage width of about four.

CONCLUSIONS

A comparison of seven forms of tension test specimens, using a wide variety of low-carbon sheet steel of various grades and thicknesses, indicates that the yield point and tensile strength are not appreciably affected by varying the gage length, width or thickness.

The percentage of elongation, however, varies appreciably with the gage length and cross-sectional area of the specimen.

The conclusions of other investigators as to the effects of these variables have been confirmed.

The precision of the measurements indicates that the gage length should not be much less than 2 in. and that the length preferably should be four times the width.

The specimen suggested by Committee E-1 on Methods of Testing of this Society for several reasons other than those covered by this paper, with a gage length of 2 in. and a gage width of $\frac{1}{2}$ in., gave on the whole the most satisfactory results, and should be suitable for use in testing low-carbon sheet steel.

Acknowledgments.—The authors wish to acknowledge the assistance of the staff of the Research Laboratory, particularly Messrs. M. E. Hazlett, J. O. Keighley and A. G. Young.

THE APPLICATION OF MAGNETICS TO THE INSPECTION OF STEAM TURBINE BUCKET WHEELS

By J. A. Capp[1]

Synopsis

Apparatus for testing steam turbine bucket wheels is described, by which, without destroying the wheel, the homogeneity and soundness of the steel may be quite accurately determined. The method of testing consists in the indication of the presence of defects by changes which they cause in the reluctance of a magnetic circuit of which the wheel under test is the only variable part. The indications are obtained by the use of a high-sensitivity galvanometer connected to measuring coils located in the specially designed pole-tips of the electro-magnet between which the wheel is rotated. Typical charts of tests are given and photographs are shown of typical hidden defects located by the apparatus.

The modern steam turbine may operate at speeds up to 3600 r.p.m. and deliver power up to 90,000 kw. and more. The proportioning and dimensioning of the component parts of such huge engines has become a highly developed art, but the art of engineering design must have, as its basis, available materials which may be depended upon to have the required properties. Dependability is evidently a requisite which can hardly be over-emphasized, and nowhere is it more necessary than in the bucket wheel. Small test specimens may be taken from a number of places in a wheel and give one a very good survey of its composition and strength. Macroscopic and microscopic examination will yield valuable information as to its structure and treatment. Visual examination of the wheel as delivered, and as machining operations reduce it to its final form and dimensions, gives an idea of its soundness and sometimes may enable one to cull out an obviously defective piece of steel. Yet all these usual means of testing and inspection leave one without information on the homogeneity and soundness of the actual wheel itself. The company with which the author is connected felt that it was desirable to supply this lack to the greatest extent possible, and means to this end were sought. Examination by means of the X-ray was considered but the large size of the objects to be examined made the X-ray impractical. A bucket wheel may be as large as 10 ft. in diameter. The testing of the wheel by magnetic means seemed to give the greatest promise of success and the development of this method of testing was decided upon. To help in the preliminary work, the services of the late Dr. Chas. W. Burrows were secured. Dr. Burrows had made the study of magnetics his life work and had already had considerable success in solving problems of a similar nature, though even he had not attacked the routine testing of any object as large as a turbine wheel.

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[1] Chief of Testing Laboratory, General Electric Co., Schenectady, N. Y.

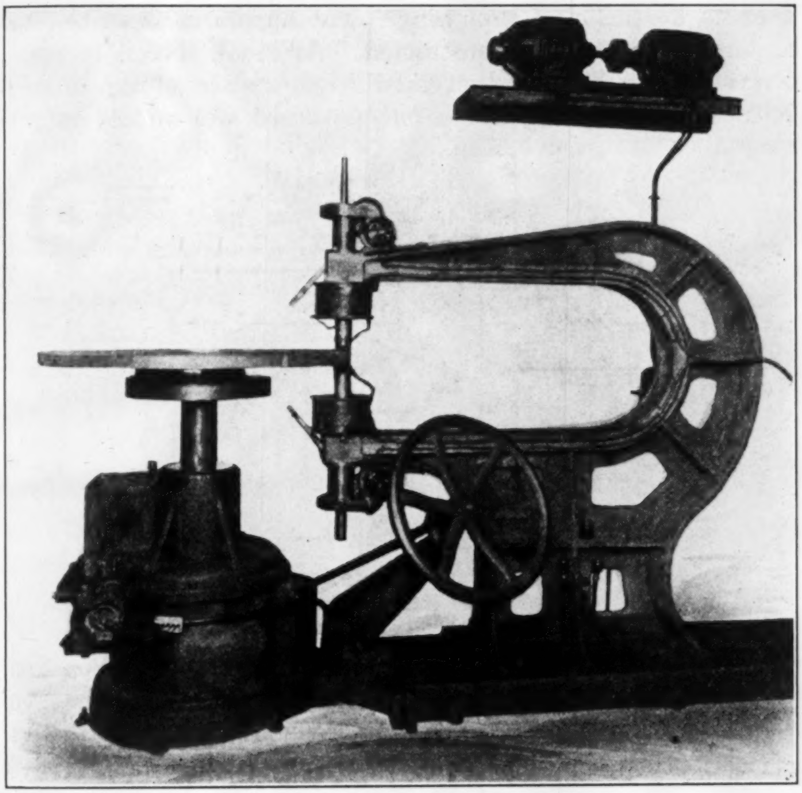

FIG. 1.—Magnetic Flaw Detector.

The steam turbine bucket wheel lends itself readily to magnetic testing because of its form. It is essentially a disk, tapering in thickness from its hub to the enlargement at the rim, where the buckets are attached, and is designed to rotate in a plane at right angles to the axis of the shaft. The most obvious method of testing was to rotate the wheel between the poles of an electro-magnet so that differences in parts of the wheel might be indicated by measurable differences in flux passing from pole to pole through the wheel. A number of other methods of magnetic testing were considered but the method mentioned was selected as the most easily practicable one, and it seemed to be the most promising. The apparatus described was accordingly designed and constructed. As usual, success in operation was not immediate, and several different schemes of measurement were tried before the very simple one, now used, was worked out.

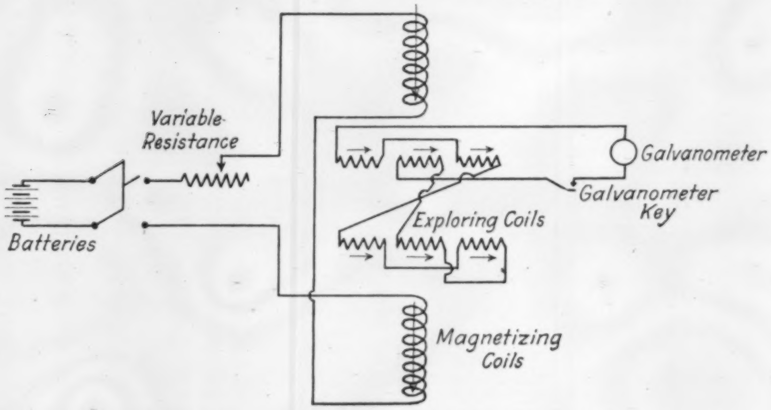

FIG. 2.—Schematic Diagram of Wiring for Magnetic Analysis of Turbine Bucket Wheels.

APPARATUS AND METHOD OF OPERATION

Figure 1 shows the apparatus with a bucket wheel set up for testing. It consists essentially of a vertical spindle carrying, at its upper end, a chuck by which the wheel is held and turned between the poles of the electro-magnet which consists of the yoke spanning the wheel and carrying cylindrical poles upon which the energizing coils are mounted and in the faces of which measuring coils are placed. The energizing coils are connected to a source of current and the measuring coils are connected to a galvanometer. The yoke is mounted upon V rails on which it may be moved back and forth, thereby shifting the poles radially over the wheel. The poles are adjustable to the thickness of the wheel, and attachments are provided for extending the poles so that one may project into the bore of the wheel or over its edge. By these means, all parts of the wheel from the hub, through the web to the rim may be brought between the poles and subjected to test. The details of the mechanical drive of the spindle and the means of shifting the yoke are omitted as they are not essential to an understanding of the operation of the device.

The electrical connections are shown diagrammatically in Fig. 2 and the construction of the pole-tips, with the placing of the measuring windings, is shown in Fig. 3. It is evident that the energizing coils may be connected so that the current will flow in the same direction in each, thereby making the one pole North and the other pole South. This connection will produce a flow of magnetic flux from one pole through the air gaps and wheel to the other pole and then around the yoke to the first, in one continuous circuit. Or the circuit may be reversed in direction through the two energizing coils, making them both North or South, and hence bucking each other. With this connection, the flux flows from both poles toward the wheel and immediately spreads out in the plane of the wheel. Experience has shown that best results are obtained with the poles acting in series and the tests are made with these connections.

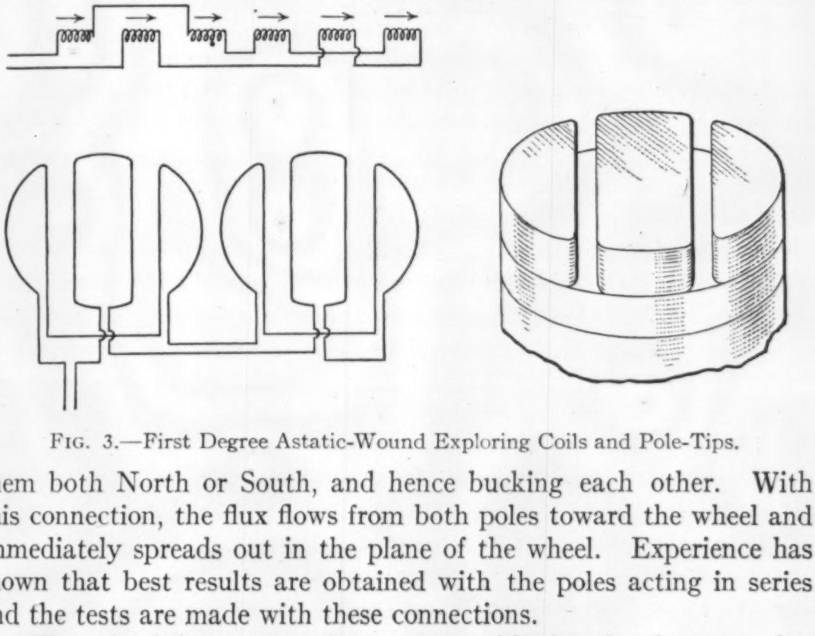

FIG. 3.—First Degree Astatic-Wound Exploring Coils and Pole-Tips.

The pole-tips are grooved so as to provide slots for the measuring coils which are in three sections, connected astatically so as to negative the effect of eddy currents set up in the wheel as it rotates. The measuring coils, connected as shown in Fig. 3, are then connected to the galvanometers, of which there are two. One is mounted in the usual way for reading its deflections on a transparent scale and is used for visual observation. The other is mounted in a box which contains the recording mechanism. The indicating light line from the galvanometer mirror passes through a slit and falls on sensitized paper which is mounted on a disk. This disk is rotated in synchronism with the wheel under test, and its center is movable to correspond with the shifting of the yoke frame of the machine. There is thereby recorded on the sensitized photographic paper a roughly circular line whose radius is proportional to the radius of the part of the wheel between the pole pieces.

In the test, a ring element of the wheel passes between the pole faces and the current in the energizing coils is held constant so as to set up a constant magnetomotive force. The wheel is machined to a very great accuracy of profile, hence, the ring element passing between the poles varies only very slightly in thickness, and the air gap is of corresponding uniformity. The poles, yoke frame and air gap, with the wheel, constitute the magnetic circuit. The magnetic reluctance of the poles and yoke frame is fixed and that of the air gap will not vary so long as the gap is uniform in length. Hence, if the wheel is magnetically homogeneous and uniform in thickness, the magnetic reluctance will remain constant as the wheel rotates, and there will be maintained a uniform flow of magnetic flux. If the reluctance of any part of the circuit changes, there will be a corresponding change in flux which will be indicated by the galvanometer. It is evident that if there is a constant flux between the pole-tips, there will be no current flow in the measuring coils, while any change in flux will set up a current in the measuring coil and cause the galvanometer to deflect. The galvanometer and its circuit may be adjusted to measure the change in flux, whether increasing or decreasing. From what has preceded, it should be evident that the only part of the magnetic circuit which may be expected to vary is the wheel under test.

The many papers on the general subject of magnetic analysis and especially papers of Doctor Burrows have shown that any substantial change in the constitution or state of steel will be reflected in a corresponding change in its magnetic characteristics. Conversely, observed changes in magnetic characteristics may be accepted as indicating changes in constitution or state of the steel. Advantage has been taken of this principle in the development of the method of testing under discussion. Taking any given bucket wheel, we have an apparatus capable of measuring, or, at least of indicating any difference which may exist in different parts of any ring element of the wheel as it is rotated between the poles of the machine. For example, it may be assumed that somewhere in the web of the wheel, there is a hard spot. The uniform parts of the ring element containing the hard spot will be of practically uniform permeability, while the permeability of the hard spot will be materially less. When the ring element is rotated between the poles of the yoke, there will be a sharp increase in the reluctance of the magnetic circuit as the hard spot passes between the poles. This increase of reluctance causes a decrease in total flux, and the change in flux sets up a current in the measuring coils thereby causing the galvanometer to deflect. As the hard spot passes out from between the poles, the reverse change takes place and the galvanometer deflects in the opposite direction. If care be taken to correlate the indications of the galvanometer with the exact spot on the wheel passing between the poles and out again, the hard spot may be located within very narrow limits. By shifting the poles in or out on the wheel, and passing new ring elements between the poles, the area of the hard spot may be outlined. Anything which causes a localized change from the surrounding average magnetic reluctance of the steel in a wheel may be located, since it will act similarly to the hard spot taken as an illustration.

It is evident that, considering the magnetic circuit as a whole, the wheel itself is only a relatively small part of the circuit, and any change in the reluctance of the wheel will only cause a small change in the total reluctance of the entire circuit; hence, there will be only equivalently small changes in the total flux due to such changes as may occur in the reluctance of the wheel. The means of measurement must, therefore, be sensitive to such small changes, regardless of the magnitude of the total flux and of the influence of extraneous variables. This is accomplished by using an over-damped ballistic galvanometer of very great sensitivity, and by the careful construction and proportioning of the pole-tips and measuring coils. If a solid pole-tip surrounded by a single measuring coil were used, it would be possible to adjust the number of turns in the coil and the sensitiveness of the galvanometer so that any change in the reluctance of the wheel would be indicated, but there would be a nearly continuous drift of the galvanometer as the wheel is rotated. The wheel is a conductor rotating in a field of magnetic force, and eddy currents are set up in it. Just how these in turn set up inductive forces acting on the galvanometer would be an interesting study, but (without pursuing the matter deeply) it was found they do cause a drift of the galvanometer and sometimes nearly obscure the effect of relatively large changes in the wheel itself. Their effect was overcome by making the pole-tip in three sections and then winding a separate coil around each section and connecting the three coils astatically. Careful adjustment of the areas of cross-section of the three parts of the pole-tip is essential. The two outside segments must be of exactly the same area, and each must have exactly one-half the area of the central segment. The three must have, as nearly as possible, the same magnetic and electrical characteristics. They are machined from one piece of soft steel, carefully annealed. The dimensions are very carefully adjusted and measurements are equally carefully checked by weighing. Being of the same height, weight is a check on dimensions if the three pieces are otherwise identical. The coil wound on each part is of exactly the same number of turns. These are necessary precautions since any unbalance causes a drift of the galvanometer, reduces the sensitiveness of the apparatus, and renders a photographic record practically impossible.

By the use of the high-sensitivity galvanometer connected to the three coils, very small things may be detected, things so small that the indication is seemingly out of all proportion to the cause. For example, if a rough-turned wheel is tested, the galvanometer is continuously on the jump, due to the wavy surface left by the roughing tool; yet these ridges are circular and move parallel to each other as they pass between the poles. A scratch on the surface of a wheel made by a scriber drawn several times back and forth will cause a large deflection of the galvanometer as it passes between the poles. The imaginary hard spot used in an earlier paragraph as an illustration might cause the galvanometer to swing entirely off the scale. As set up for testing, use is made of both the reading and the recording galvanometers. The latter has extra resistance in circuit so as to cut down deflections to a scale that can be recorded on the photographic diagram, while the reading galvanometer will swing from 15 to 20 times as far due to the same impulse. The photographic record is a convenience for filing, while the acceptance or rejection of a wheel is determined by the record of the reading galvanometer. A suspicious spot may be critically examined at a number of different radii before a decision is reached.

ILLUSTRATIONS OF APPLICATION

Let us now consider some of the things which might cause localized changes in reluctance of the steel as part of the magnetic circuit of the apparatus and the indications that may be expected of such changes.

Bucket wheels in the modern turbine are almost exclusively forgings, but in the earlier days, castings were used, and some wheels of smaller diameters and thinner sections were machined from rolled plate. Formerly, all wheel blanks, however made, were annealed as the only heat treatment. The forgings used to-day are of plain carbon steel and of 3 to 3.5 per cent nickel steel, and both are customarily annealed and then quenched and tempered. Our experience with magnetic testing is confined almost entirely to forgings, although we have tested a number of wheels machined from rolled plate. The use of castings had practically been abandoned before the introduction of the magnetic test.

A blowhole in the ingot will usually be closed and elongated by the work of rolling. In a forging which must be upset to the disk shape required for a bucket wheel, the blowhole may be flattened and slightly spread as well as drawn out by the forging work. In neither case will it necessarily be welded tight; on the contrary, it will frequently be equivalent to a seam whose sides are close together. Such a "seam" will cause a sharp deflection as it passes the entering edge of the pole and another as it leaves the pole. It is surprising how small a "seam" can be found in a wheel web. Figure 4 shows such a defect. The strip of steel is about $\frac{5}{16}$ in. thick and was cut from the web of a wheel about $\frac{3}{4}$ in. thick. Successive machine cuts were first taken on the sides, reducing the thickness of the strip until it was only about $\frac{5}{16}$ in. thick, and still no defect had been found. The strip was of considerable width, so a series of cuts was then begun to reduce the width, and finally the seam, which was evidently a closed but unwelded blowhole, was disclosed. At the time of testing this wheel the autographic apparatus had not been installed and all readings were taken by observing the galvanometer. The galvanometer beam is never completely steady, but swings slightly because of minor changes in homogeneity of the steel and slight variations in thickness of metal. These swings are of the order of 3 or 4 cm. The "seam" shown in Fig. 4 caused a sharp kick of the galvanometer beam of 10 to 15 cm.

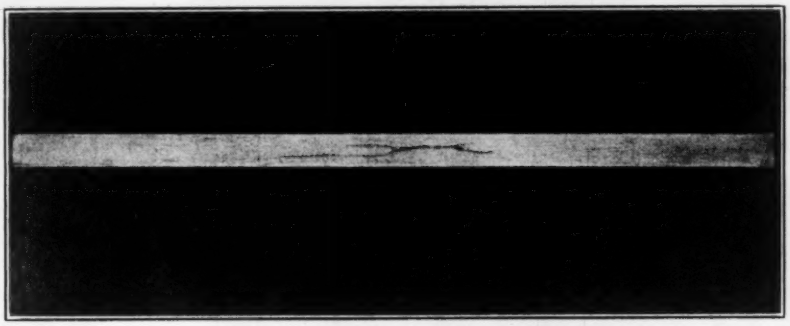

FIG. 4.—Turbine Bucket Wheel of Heat-Treated Nickel Steel Which Gave Excessive Deflections in Magnetic Test.

In upsetting a blank to the disk form, it would be possible to tear or burst the metal by injudicious hastening of the reduction of thickness. Such bursts have been found, indicated by the same type of "kick" as resulted in the case of the "blowhole seam," but the kick is usually greater.

The quenching of a wheel blank may cause cracks, especially at the rim, too small to be easily seen. They are shown up by the kick of the galvanometer, whereas sometimes a good deal of careful searching is required to find them by visual examination.

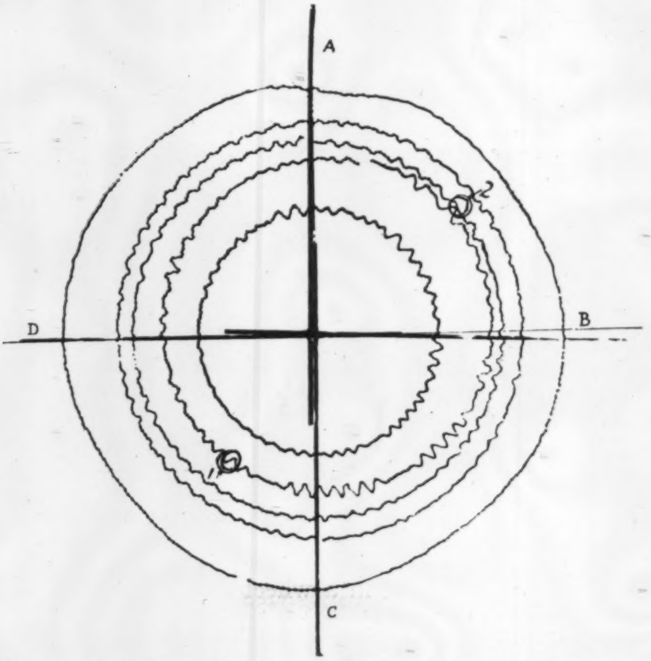

FIG. 5.—Autograph Record of Magnetic Test of Imperfect Turbine Bucket Wheel.

The manufacture of steel free from slag inclusions, manganese sulfide and the other things called non-metallic inclusions is doubtless the goal which makers of high-grade steel are seeking to attain. Notwithstanding the care taken to avoid the presence of these inclusions, concerning which there has been so much discussion, they have an annoying habit of turning up when least expected, and they occasionally exhibit a most annoying tendency to colonize much like a germ colony in an infected human, so that, while the main body of the steel may be quite clean, there will be local areas in which there are definite aggregations of these inclusions. A bucket-wheel forging is looked upon as a high-class product warranting the use of only the best in the steel-making art, and these segregated areas of inclusions are looked upon, at least, with considerable suspicion. The magnetic test is an excellent means of locating these colonies of inclusions when they exist to an extent considered to be prohibitive. Figure 5 is the autographic record of a magnetic test of a wheel. It will be noticed that with the exception of the outer circle all of the lines are wavy and show numerous sharp deflections. Two points marked 1 and 2 were selected as about the worst as indicated by the kicks and specimens were taken for examination. We may say, in passing, that the necessity for the placing of steam balancing holes in the web of the wheels frequently offers the possibility of taking specimens from a wheel without destroying it. Consultation with the engineers and factory organization frequently permits the layout of a balancing hole at the spot where the taking of a specimen is desired. By drilling small holes in a ring about the selected spot, a core section of the wheel is available for examination. This was done in the case of the wheel whose record appears as Fig. 5. A section of this core was polished and is shown, at 100 diameters without etching, in Fig. 6. For comparison, there is shown in Fig. 7 the records on the web of a wheel which may be considered typical of the general run of wheel forgings. It will be noted that the curves are practically free from breaks and especially from sharp kicks.

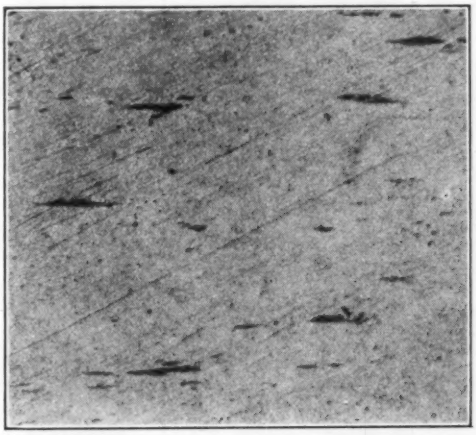

FIG. 6.—Section of Turbine Bucket Wheel Polished and Unetched. (× 100).

One more illustration may be given to show the working of the magnetic test. In Fig. 8 is shown the magnetic test record of a wheel

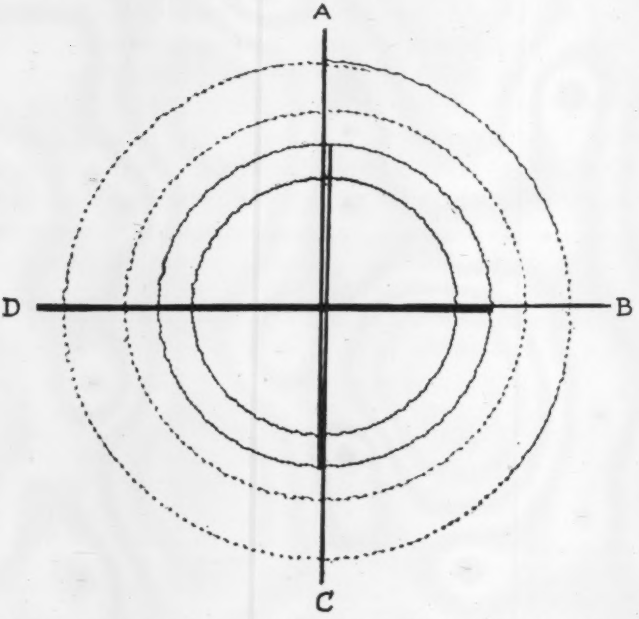

FIG. 7.—Typical Autographic Records of Turbine Bucket Wheel in Magnetic Tests.

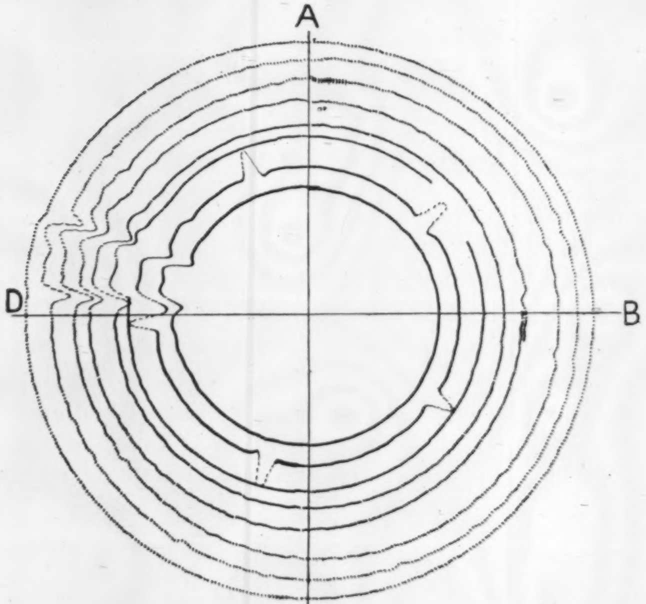

FIG. 8.—Magnetic Test Record of Turbine Bucket Wheel, Showing Effect of Steam Balancing Holes and Area Containing Inclusions.

tested after the steam-balancing holes had been placed and the wheel practically finished. The five kicks in the second curve show these balancing holes. All of the curves are very smooth except in one sector, where there are characteristic sharp deflections. Our established practice prohibited the use of this wheel, which was then cut up for examination. Figure 9 shows a block machined from about the center of the suspected sector and shows the cause of the magnetic disturbance, but does not show up the most interesting features of the specimen. At each surface is a band of very clean steel $\frac{1}{16}$ to $\frac{1}{8}$ in. thick, but between these bands the steel contains a really amazing amount of inclusions; recognizable as of a slaggy nature. This slaggy region was quite sharply defined, and the density of the inclusions was quite uniform. The case was fully investigated in cooperation with the maker of the forging, and the only conclusion possible was that a piece of the runner, used in pouring the ingot, had broken away and been carried up into the body of the ingot, where it had only diffused to a very small extent. It happened to lie in the center of the height of the slice from which the wheel was forged, and remained in the center of thickness of the wheel web. Wheels made from adjacent slices from the same ingot passed satisfactory tests.

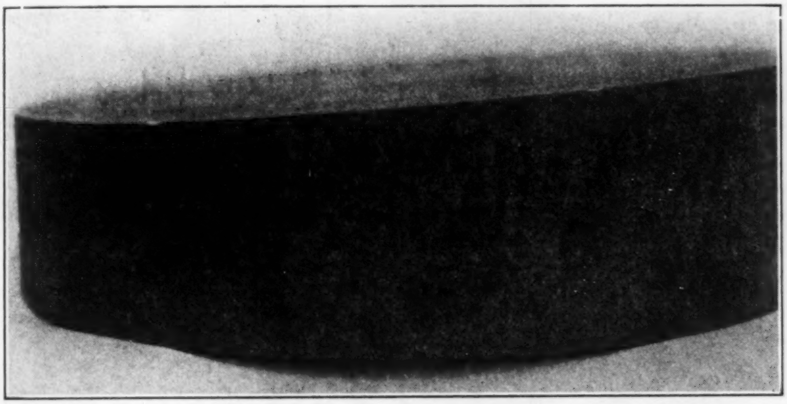

FIG. 9.—Section of Turbine Bucket Wheel Cut From Area Giving High Deflections Under Magnetic Test, Showing Inclusions.

It will be obvious that it is highly improbable that any of the ordinary methods of examination and testing would have indicated the presence of any of the defects illustrated. The illustrations are types, not isolated cases, except that of the broken runner. Notwithstanding the high standard set for bucket-wheel forgings, the manufacturers have been successful in meeting it. This is shown by the final rejection because of the results of the magnetic test of only 23 out of some 3000 wheels tested, although many more were under suspicion until the defects were removed in the process of machining.

Concerning this method of examination, it is clear that it only indicates the presence and location in a wheel, of an area of greater or less extent, where the steel differs in some respect from that surrounding the area. It does not give any definite information on the cause or character of the difference. As experience is being obtained, we find that there appear to be indications that certain sorts of defects tend to yield certain types of deflections, and other sorts of defects tend to yield other types of deflections. At present these are tendencies only, and much more experience must be gained before anything definite may be laid down as to the probable character of defect from the characteristic type of deflection noted.

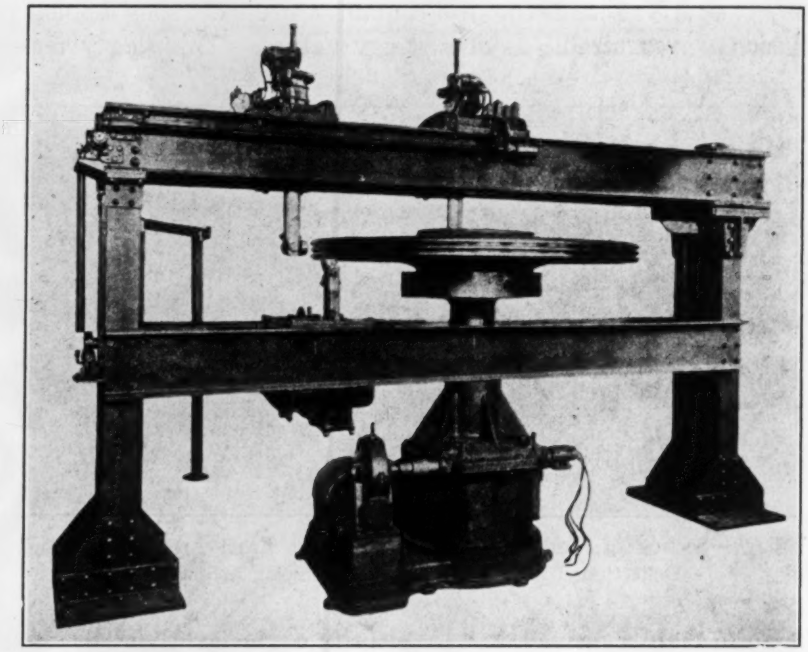

FIG. 10.—Magnetic Analyzer for Turbine Bucket Wheels (Most Recent Design).

Formerly, wheels were tested in a semi-finished state. The rough machined forging was smooth turned and then tested. Frequently, defects were indicated, and exploration of the area showed defects which would machine out in finishing the forging to size.

When so finished, the wheel would be passed by the magnetic test. Much valuable experience was gained in this way, but the cost of setting up the forging in a mill for partial machining and then setting it up again after testing was so great, in comparison with the number of wheels actually rejected, that it was obviously safe and economical to do the testing only after finishing the turning operations on the wheel. This has slowed down the gaining of experience on the correlation of defects with their indication by the magnetic test. But the experience gained has warranted us in setting a fairly fixed limit to the size of a deflection which is permissible, especially when it is considered in connection with its persistance. Deflections not exceeding 8 to 10 cm. on the observing galvanometer scale are not necessarily suspicious, unless they are persistent, when they may indicate too many inclusions. Any discontinuity will usually give a sharp deflection of more than 10 cm. In any case, the magnetic indications are followed by visual examination, even though the wheel is sacrificed to obtain the necessary specimens. It is a significant fact that we have never had a magnetic indication of trouble without finding an adequate cause. Our experience has developed confidence in the method of testing, and we feel safe in the belief that this method of examining wheels supplies the needed information on the homogeneity and soundness of the wheel itself after all the other means of testing have given the usual information on its qualities, treatment and structure.

DISCUSSION

Mr. Lynch.

THE CHAIRMAN (*Mr. T. D. Lynch*[1]).—In this day of central power stations, when we are trying to get all we can out of fuel, one can realize what this test described by Mr. Capp means. Mr. Capp has shown a most interesting method and type of testing. It is very important to know that the material is right. Turbine wheels operating at the terrific speed of eight and one-half miles per minute are under very high stresses.

There is one further thought that I should like to introduce and that is the question of temperature and strength of materials at these temperatures. These buckets run by steam at superheat temperature and the high temperature reduces the strength of the material, so that it is all the more important to know that the material is correct.

This subject is so interesting that I am sure there are some here who would like to discuss it and ask Mr. Capp questions. The paper is open for discussion.

A Member.

A MEMBER.—I should like to ask Mr. Capp a question. Suppose that the wheel were very uniformly dirty—defective, in other words; what would then happen as it passed slowly between the poles? Would there be any kicks? I should imagine that there would be no kicks in the curve. Where then is the comparison?

Mr. Capp.

MR. J. A. CAPP.[2]—I believe that if the steel were uniformly dirty, it would probably not give a kick; but I have never seen any steel that was quite so uniformly dirty as that. It is a fortunate fact that when a steel is slaggy, if you want to use that term, it is not uniformly slaggy. The distribution of non-metallic inclusions is irregular, and the irregularity will inevitably show up. If it were perfectly uniform as to the distribution of internal defects, we would not find them.

A Member.

A MEMBER.—I should think that if one were using basic electric steel, one might happen to find that condition. If one were using an open-hearth steel, I agree with Mr. Capp that one would get a kick if the steel were dirty.

Mr. Capp.

MR. CAPP.—The wheel forgings are large; they weigh in the rough as high as 20,000 lb. and more. They are always made of open-hearth steel. They are usually made from relatively large ingots, which means that they are made from large melts. A wheel of 8 or 10 ft.

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[1] Manager, Materials and Process Engineering Department, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] Chief of Testing Laboratory, General Electric Co., Schenectady, N. Y.

diameter, for instance, would probably not be made from a 12 or even an 18-in. ingot, but from at least a 36-in. ingot, because the amount of work necessary to flatten out the disk or slice cut from the ingot would be too great, and there would be too much liability of tearing at the periphery before it had been worked out to the necessary diameter. The hub may be 12 in. high, while the wheel rim thickness may be a little over 2 in. at the outer edge, so it takes a big ingot; and that means, as most plants are rigged up today, open-hearth steel. **Mr. Capp.**

MR. J. A. REINHARDT.[1]—I should like to ask Mr. Capp what effect the gradual changes in thickness between the pole pieces would have? He mentioned that slight scratches had a great effect on the kick in the galvanometer. He also mentioned that there is a variation in thickness from the outside to the inside of the rim of this wheel. Does he have to adjust for that change in thickness? **Mr. Reinhardt.**

MR. CAPP.—Yes. When we select any particular ring element, one just inside the rim, for instance, the pole pieces, which are adjustable, are set to the proper air gap. As I recall it, it is of the order of $\frac{1}{4}$ in. The first ring is run around and tested—more than once if necessary. The pole pieces are then shifted inward, one width of the pole, about 3 in. on the wheel. If there is a change in thickness of the wheel, adjustment of the air gap is made. We do not run the poles rapidly on the wheel while testing. **Mr. Capp.**

MR. THOMAS SPOONER.[2]—I should like to ask Mr. Capp what he finds to be the most suitable air gap induction for this type of test. **Mr. Spooner.**

MR. CAPP.—It is relatively difficult to measure what is the flux passing. I do not know that I have any figures even indicating what it is. The steel used is mostly a 3-per-cent nickel steel, quenched and tempered. In the metal at the rim, which is never over 4 in. thick, we obtain normally about a 70,000 lb. per sq. in. proportional limit, with in the neighborhood of 100,000 lb. per sq. in. tensile strength. Probably Mr. Spooner knows what the induction curve of such metal is. Our measurements indicate that we are just about on the knee of the curve. **Mr. Capp.**

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[1] Metallurgist and Assistant Chief Engineer, Western Clock Co., La Salle, Ill.

[2] Research Engineer, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

SOME USEFUL APPLICATIONS OF A QUARTZ SPECTROGRAPH

By F. A. Hull[1] and G. J. Steele[2]

Synopsis

The paper discusses briefly some useful applications of a quartz spectrograph both as regards qualitative and, in a sense, quantitative analysis. Particular emphasis is laid on the great economy resulting from savings in the analyst's time and especially in inventory. Present limitations of methods of quantitative examination are discussed and suggestions made as to direction of future work.

Some years ago we ran into a very serious difficulty in the production of certain X-ray tubes due to the formation of what seemed to be a metallic mirror in one end of some of the tubes. Neither the copper anodes nor the deposit showed anything out of the ordinary by customary analytical means. As a last resort, some of the tubes were sent to the analytical control laboratory of a large industrial plant where there was installed and in operation a Hilger spectrograph. Examination then showed the presence of minute traces of zinc on the surface of the copper anodes. With this clue, it was soon found that an over-zealous workman, in attempting to turn out a highly finished anode, had used a buffing wheel which had previously been used to polish brass. There was no further trouble with mirrors on the X-ray tubes.

With this incentive we were led to look into the general usefulness of the spectrograph, secured one, and a few of the numerous applications in which the spectrograph has been of service are now presented, in the hope of eliciting comment, creating greater interest, and promoting still greater usefulness.

Brief Description of Spectrograph

The excellent papers by Meggers[3] and by Bassett and Davis[4] have previously described the instrument and its operation. Beyond showing photographs of the instrument as set up in our laboratory, we will not take up time to enter into details of this phase of the subject.

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[1] Chief Chemist, Testing Laboratory, General Electric Co., Schenectady, N. Y.

[2] Testing Laboratory, General Electric Co., Schenectady, N. Y.

[3] W. T. Meggers, C. C. Kiess and F. J. Stimson, "Practical Spectrographic Analysis," U. S. Bureau of Standards *Scientific Paper No. 444*.

[4] W. H. Bassett and C. H. Davis, "Spectrum Analysis in an Industrial Laboratory," paper presented before Am. Inst. Mining and Metallurgical Engrs., No. 1134-N, February, 1922.

Figure 1 shows the instrument mounted on a firm table, subject to little or no vibration. The exhaust hood and blower necessary to carry off various fumes which might be detrimental to the operator are also shown. Figure 2 is a view of the instrument from the other end, and shows the photographic attachment and the cabinet used to house the sparking and arcing apparatus, which are not furnished with the spectrograph. Attention is called to the window fitted with cobalt glass to stop ultra-violet rays from affecting the operators.

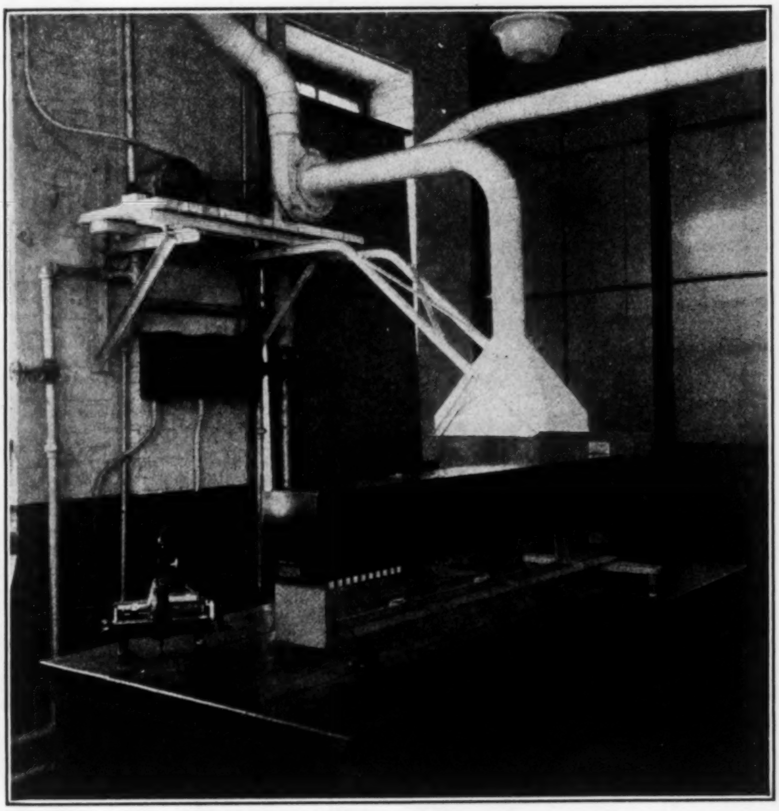


FIG. 1.—The Spectrograph Mounted.

Figure 3 shows the sparking and arcing apparatus. The electrodes, consisting generally of the sample to be "sparked," the quartz condensing lens, and the switch which permits of operation only when the door is closed, are particularly to be noted.

The electrical part of the quartz spectrograph consists of (1) a direct-current supply with regulating resistance for producing a direct-current arc through the sample electrodes, and (2) an alternating current supply with transformer, condensers, and coils for producing a high-frequency alternating current spark between the sample electrodes. The wiring diagram is shown in Fig. 4.

When the main switch (6-pole double-throw) is closed on the side marked *d. c.* and the protective switches are closed, the circuit is arranged for the direct-current arc and the current path is substantially as follows: from the positive terminal through the switches to the ammeter, through the gap and regulating rheostat back to the negative terminal of the supply. The usual voltage is 115 and the current is 3 to 5 amperes, depending on the nature of the electrodes.

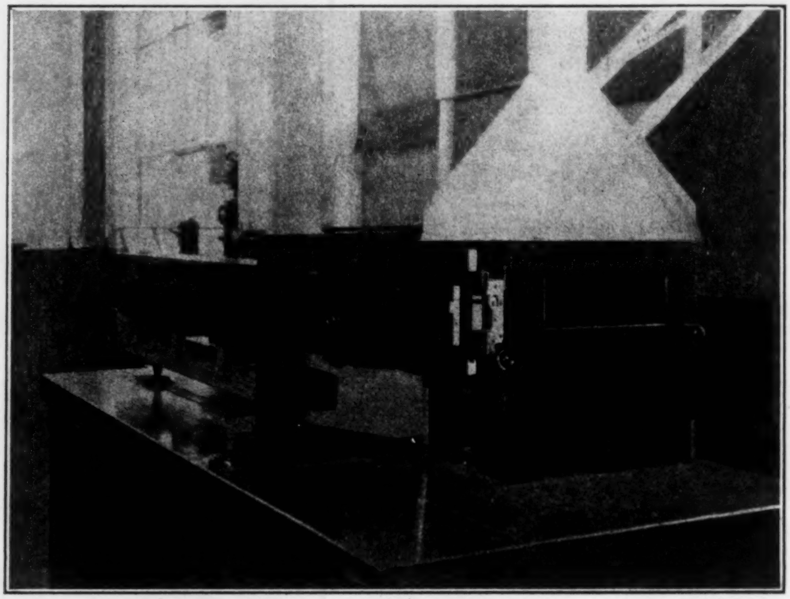


FIG. 2.—The Spectrograph Showing the Photographic Attachment and Cabinet for Housing the Sparking and Arcing Apparatus.

When the main switch is closed on the side marked *a.c.*, and the protective switches are closed, the circuit is arranged for the alternating current spark test. The action is as follows: The supply current, in this case 60-cycle 115-volt, flows through the primary of the transformer and then through a current-limiting reactor. The transformer has step-up ratios from about 20 to 1 to 50 to 1 and the secondary voltage may be changed by the single-blade rotating switch from 2000 to 5000 volts in 1000-volt steps. It is ordinarily used at the highest voltage. This high voltage is connected across an 0.04-microfarad condenser (rated at 15,000 volts for safety factor). Across this condenser is connected a series circuit of another 0.04-microfarad condenser, the spark gap and meter and the variable inductance coil.

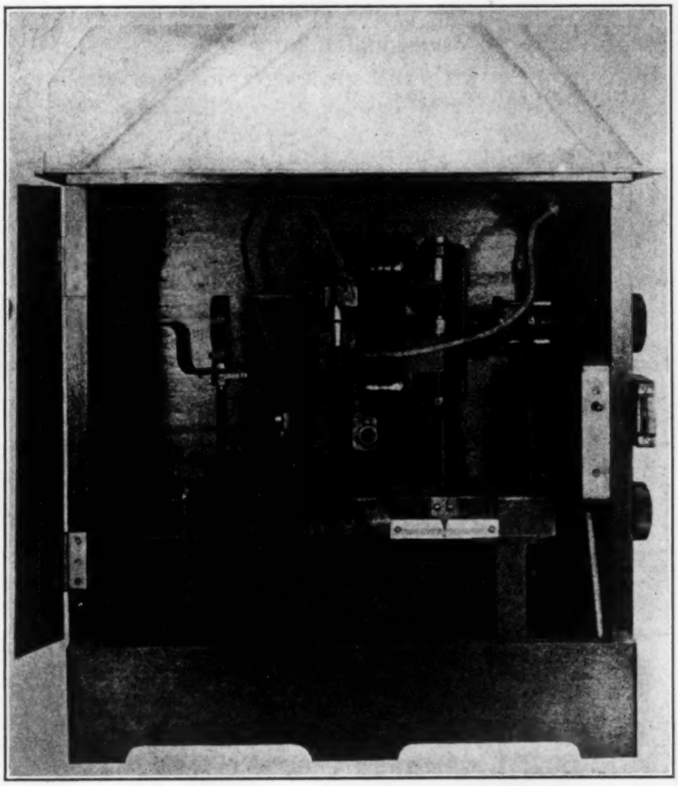


FIG. 3.—Sparking and Arcing Apparatus. Electrode Holders for Spectrograph.

This arrangement makes an ordinary type of high-frequency oscillator of the set and keeps the low-frequency 60-cycle current out of the spark gap. This prevents overloading the transformer and keeps the current in the spark gap more nearly constant.

The frequency of the current in the spark gap is about 146,000 cycles per second and the value of inductance commonly used is 59 microhenries. The usual current used is 4 to 6 amperes. The electrodes vary from $\frac{3}{16}$ to $\frac{13}{32}$ in. in diameter.

APPLICATIONS OF SPECTROGRAPH

The applications of the spectrograph naturally group themselves into qualitative and quantitative classes. The qualitative is the older and more generally investigated field.

Qualitative Applications:

In nearly all industrial plants of any size, with diversified products, we naturally find occasional mixups in stock. Differentiation is comparatively easy with the spectrograph. For example, Fig. 5 is a spectrogram illustrating (1) ordinary pig aluminum, (2) an aluminum alloy containing 8.7 per cent copper, and 2.00 per cent zinc, (3) an aluminum alloy containing 0.5 per cent magnesium and 0.50 per cent manganese, and (4) an aluminum alloy containing 13.5 per cent silicon. The lines in the spectrogram showing the different elements have been clearly marked and the differentiation between these materials in stock is comparatively easy by this means.

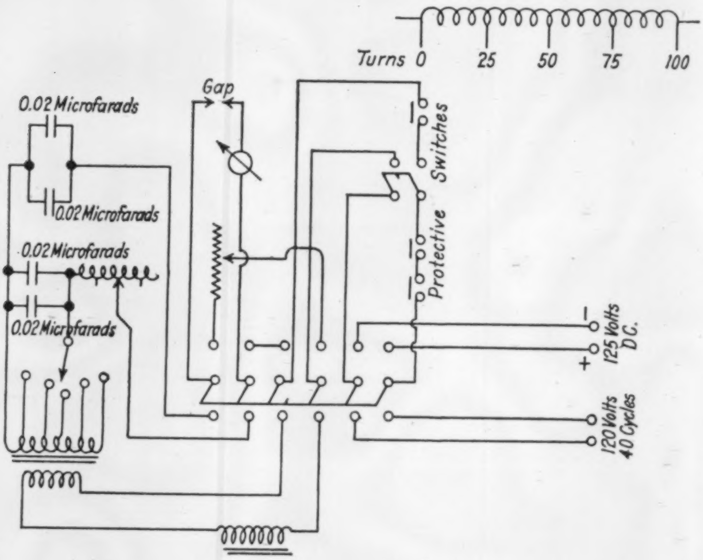
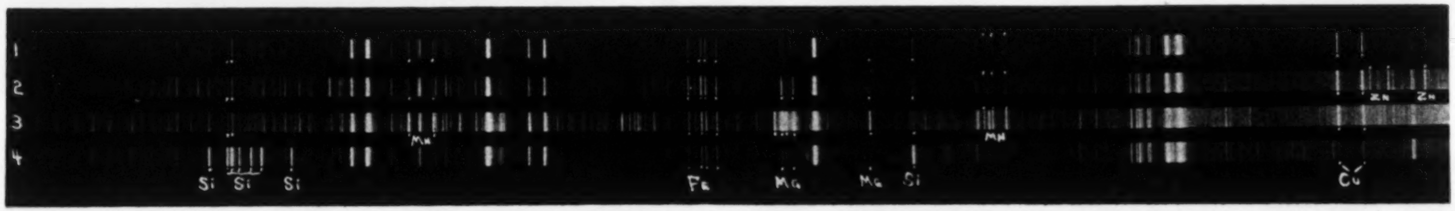


FIG. 4.—Wiring Diagram for Quartz Spectrograph Oscillator.

Frequency 146 Kilocycles. Effective Capacity in Tuned Circuit, 0.02 microfarads. Effective Inductance in Tuned circuits, 59 + microhenries. Available Inductance, 100 microhenries total, on a 2-in. Diameter Tube.

The range of the spectrograph using quartz prisms and lenses lies roughly between 2100 and 5000 Ångstrom units, while the lower limit of the visible spectrum is roughly 4000 Ångstrom units. In determining the position of the lines which will identify the elements


1
2
3
4
Si
Si
Si
Mn
Fe
Mg
Mg
Si
Mn
Cu
Zn
Zn

(1) Pig Aluminum	
Cu	0.19 per cent
Fe	0.65 "
Si	0.35 "
Mn	0.08 "

(2) Aluminum Alloy	
Cu	8.70 per cent
Fe	1.50 "
Si	0.55 "
Mn	0.06 "
Zn	2.00 "

(3) Duralumin	
Cu	3.80 per cent
Fe	0.40 "
Si	0.28 "
Mn	0.50 "
Mg	0.50 "

(4) "Alpax," Aluminum Alloy	
Cu	trace
Fe	0.40 per cent
Si	13.50 "
Mn	trace

FIG. 5.—Qualitative Analysis Spectrogram of Pig Aluminum and Aluminum Alloys.

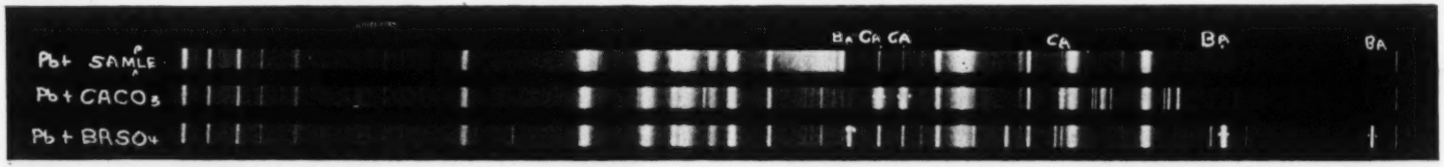

Pb + SAMPLE
Pb + CACO3
Pb + BASO4
BA CA CA
CA
BA
BA

FIG. 6.—Spectrograms of White Lead, Calcium Carbonate and Barium Sulfate.

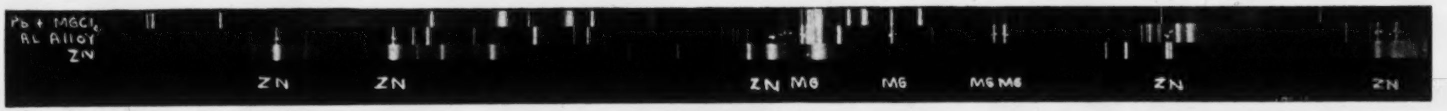
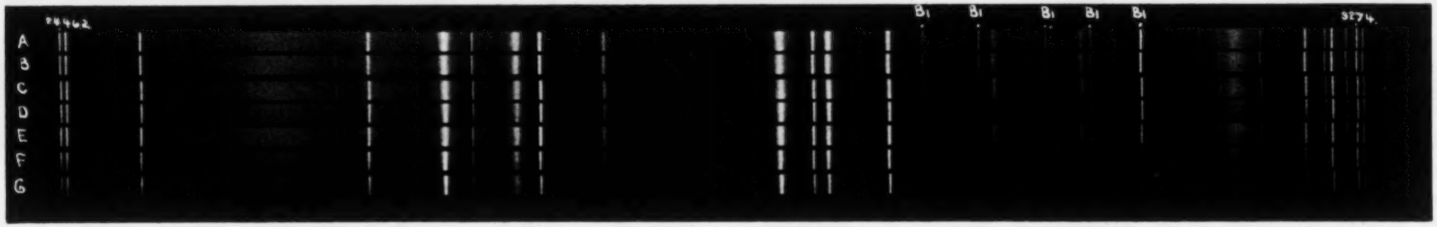
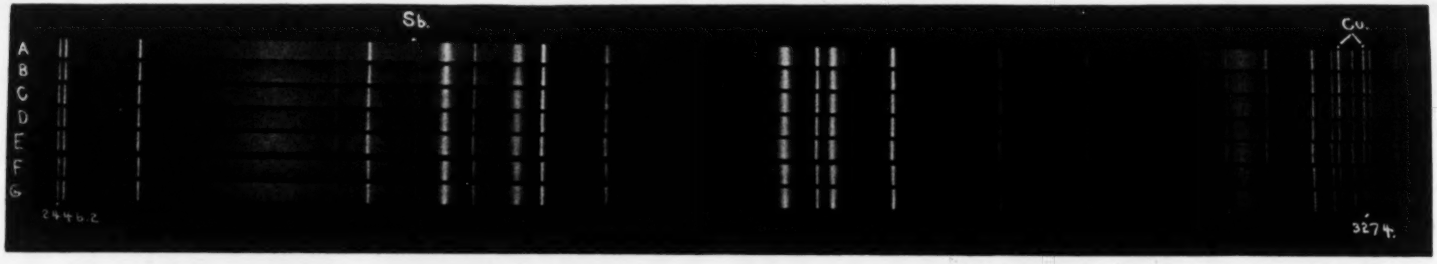


FIG. 7.—Spectrogram of Aluminum Alloy. Spectrograms of a Magnesium Salt and of Zinc are Also Shown for Purposes of Illustration.



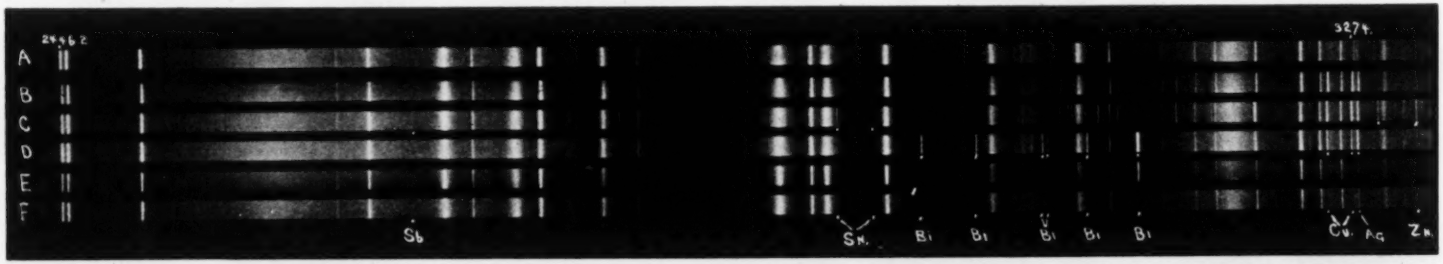
	A	*B*	*C*	*D*	*E*	*F*	*G*
Bi, per cent	0.55	0.45	0.35	0.25	0.15	0.10	0.05

FIG. 8.—Spectrograms of Pig Leads Containing Different Percentages of Bismuth.


Sb.
Cu.
A
B
C
D
E
F
G
2446.2
3274.

	A	*B*	*C*	*D*	*E*	*F*	*G*
Cu, per cent	0.08	0.06	0.04	0.02	0.007	0.004	0.0025
Sb, per cent	0.08	0.06	0.04	0.02	0.007	0.004	0.0025

FIG. 9.—Spectrograms of Pig Leads Having Various Percentages of Antimony and Copper.


2446.2
3274.
A
B
C
D
E
F
Sb
Sn.
Bi
Bi
Bi
Bi
Bi
Cu.
Ag
Zn.

A. Spectrographically Pure Lead

B. Stock Sample
Cu, 0.08 per cent

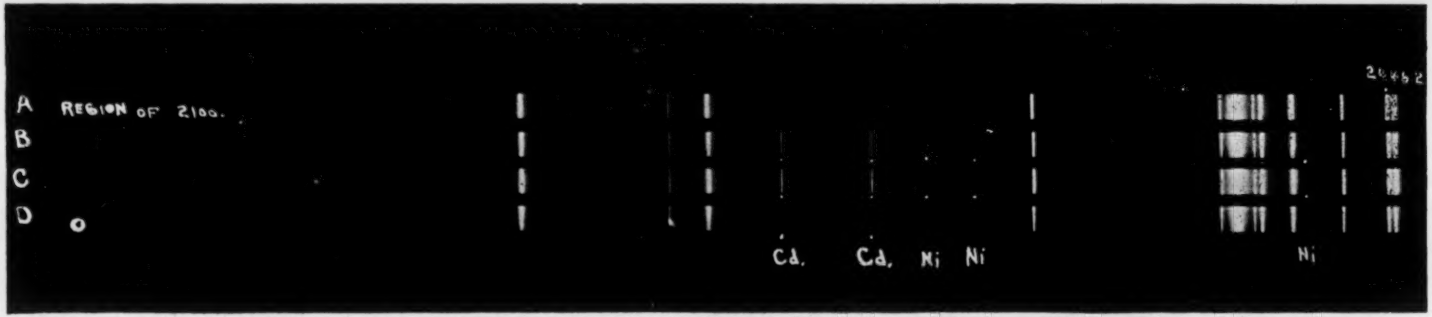
C
Cu, per cent. . 0.08
Sb, per cent. . 0.08
Sn, per cent. . 0.08
Zn, per cent. . 0.08

D. Standard Specification
Cu, per cent. . 0.0025
Sb, per cent. . 0.007
Sn, per cent. . 0.007
Ag, per cent. . 0.002
Bi, per cent. . 0.25

E. Stock Sample Within Specification

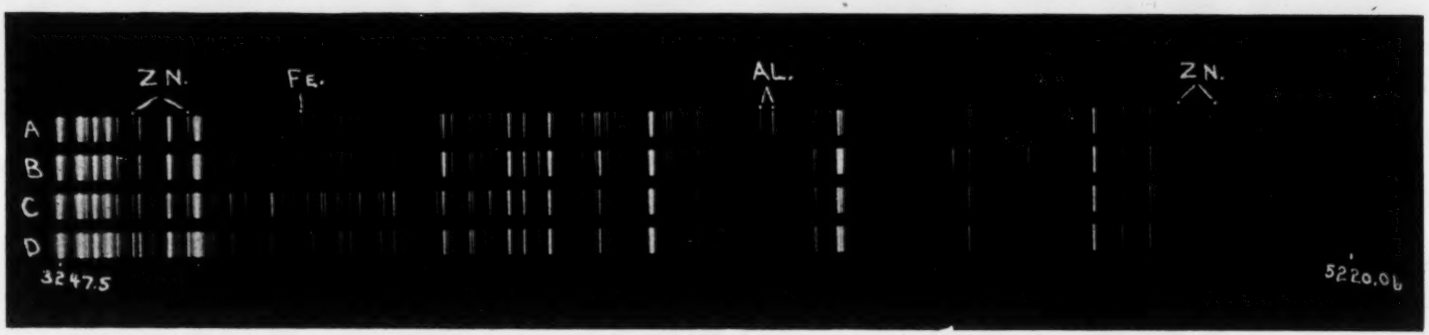
F. Standard
Zn, 0.015 per cent

FIG. 10.—Spectrograms of Six Samples of Pig Lead.


A
B
C
D
REGION OF 2100.
o
Cd.
Cd.
Ni
Ni
Ni
2446.2

A. Spectrographically Pure Lead	B. Standard	C. Stock Sample	D. Stock Sample
	Cd, per cent. . 0.01	Cd, per cent. . over specification	Cd, per cent. . . . within specification.
	Ni, per cent. . 0.01	Ni, per cent. . . " "	Ni, per cent. . . . " "

FIG. 11.—Spectrograms of Four Samples of Pig Lead.


ZN.
FE.
AL.
ZN.
A
B
C
D
3247.5
5220.06

A. Standard	B	C	D
Zn, per cent. . 0.08	Within Specification	Zn, per cent. . . 0.11	Zn, per cent. . . 0.22
Fe, per cent. . 0.12		Fe, per cent. . . Within Specification	Fe, per cent. . . Within Specification
Al, per cent. . 0.10		Al, per cent. . . Within Specification	Al, per cent. . . Within Specification

FIG. 12.—Spectrograms of Four Babbitt Metals.

present, a spectrogram of copper or iron is taken in juxtaposition to the sample under investigation. By the use of the comparator, a base line of known wave length on the copper or iron close to the line on the sample in question is chosen and a measurement made; then with the aid of a specially-prepared graph, it is comparatively easy to arrive at the wave length of the line in question. Or two iron lines of known wave length may be chosen, quite close to, and with one on either side of, the line in question, and the wave length in question computed by interpolation. In actual practice the negative is compared against one of a similar nature by superimposition, which identifies the line immediately. This is the usual procedure adopted, although a comparator should form a part of the equipment.

The examination of white lead for impurities is customarily drawn out over several days. By means of the spectrograph, a report can be made within an hour. In dealing with materials of this nature, it is obvious that no electrodes can be prepared. We are therefore accustomed to using lead electrodes, the lower one of which is hollowed out to receive some of the material in question. The resulting picture shows of course the lead spectrum plus the spectrum of the sample. Naturally, in white lead, this is of small consequence, and in the case of other materials of little more, since, in those cases, the spectrum of the electrode is always taken separately and shown in juxtaposition to that of the sample. Figure 6 shows spectrograms of a sample of white lead, of calcium carbonate and of barium sulfate. It will be readily noted that the calcium and barium lines are so faint in the sample as to justify the report of "traces," which one may normally expect.

In preparing for the wet analysis of many alloys whose elemental composition is uncertain, it is frequently of inestimable importance for the analyst to know such composition in planning the quantitative wet-analysis method. Knowledge of the absence of elements is just as important for the analyst as knowledge of their presence.

Figure 7 shows an aluminum alloy whose spectrogram shows the presence of magnesium and zinc. The spectrograms of a magnesium salt and of zinc are shown at the same time, merely for purposes of illustration, since reference to iron lines will enable the experienced operator to locate the wave lengths.

Quantitative Applications:

While the instrument is almost invaluable for purposes of qualitative analysis, its most fascinating and at the same time most valuable application has been in the field of control analysis. We wish we might unqualifiedly say quantitative analysis, but such a stage of development in application has not been reached, except where the impurities range in the neighborhood of 0.003 to 0.25 per cent, where we believe that quantitative estimation has been established with quite a number of the elements.

It is a demonstrated fact that up to a given percentage, varying with each element, the intensity of the lines in the spectrum of that element increases with increase in percentage, although not necessarily in arithmetical ratio. Beyond a certain point, differing with each element, the increase in intensity of the lines with increase in percentage of the element cannot well be measured, since the intensity apparently reaches a maximum at this certain point. This point, however, is sufficiently high in some cases to enable the operator to control the amount of impurities present in a given material where a maximum limit for such impurities has been established. We quote from F. Twyman, manager of Adam Hilger, Ltd., in a letter of February 10, 1927, to us:

> "As a result of spectrographic investigations which have been carried out in these laboratories with various metals, it may be stated that, in general, one can distinguish between 0.001, 0.01, 0.1, and 1 per cent of any metallic impurity and in many cases intermediate stages by visual estimations of the comparative intensities of the lines. The sensitive lines of de Gramont are of much use in this work, but as the percentage of impurity increases, other lines than those sensitive lines begin to appear, showing a systematic increase in intensity as the amount of impurity increases. For percentages other than the very smallest such lines not classed by de Gramont as "sensibles" are often the most sensitively variable with the percentage. The accuracy of the quantitative measurement of a percentage in a particular range depends therefore on selecting those lines which most rapidly vary in intensity with the content. Since these have not been generally investigated one must usually find them out for oneself and it is in this direction that we have been working in the specific problems which have been put up to us.
>
> "These lines are readily identified by photographing a comparison spectrum of the impurity itself. In working out routine systems of estimation, lines due to the impurity are compared with lines on the same plate due to the main bulk of the metal. This very natural precaution is advocated by W. Gerlach ("Zur Frage der richtigen Ausfuhrung und Deutung der 'quantitativen Spektralanalyse,'" *Zeitschrift für anorganische und allgemeine Chemie*, Band 142, Heft 3 und 4, pp. 383–398). He says (translation): 'Near a spectrum line λB of the substance B occur three lines of substance A, $\lambda_1 A$, $\lambda_2 A$ and $\lambda_3 A$. Adopting these as standards one finds that the intensity of the λB line,
>
> at 1 per cent B, is equal to $\lambda_1 A$
> " 0.2 " " B, " " " $\lambda_2 A$
> " 0.05 " " B, " " " $\lambda_3 A$
>
> Then in the analysis of the substance A plus x per cent B, the quantity x is determined from the brightness of the B line relative to $\lambda_1 A$, $\lambda_2 A$, $\lambda_3 A$.'
>
> "The advantage of this method is that errors due to photographic effects, such as exposure, development, etc., are obviated."

In Fig. 8 are shown spectrograms of seven different pig leads containing different percentages of bismuth. The percentages are as follows:

	Bismuth, per cent
A	0.55
B	0.45
C	0.35
D	0.25
E	0.15
F	0.10
G	0.05

It will be noted that not all the bismuth lines are of the same intensity for a given sample and that in fact some of the lines only begin to appear when the element under observation reaches a certain percentage.

Figure 9 illustrates the lines corresponding to varying percentages of antimony and copper in samples of pig lead, as follows:

	Copper, per cent	Antimony, per cent
A	0.08	0.08
B	0.06	0.06
C	0.04	0.04
D	0.02	0.02
E	0.007	0.007
F	0.004	0.004
G	0.0025	0.0025

In Fig. 10 are shown spectrograms of six samples of pig leads. Sample *A* is spectrographically pure lead, hence the lines appearing are those of lead only. Sample *D* was made to meet A.S.T.M. Specifications and had the following composition:

Copper	0.0025	per cent
Antimony	0.007	"
Tin	0.007	"
Silver	0.002	"
Bismuth	0.25	"

Samples *B* and *E* represent material as received from the refiners, sample *B* containing 0.08 per cent copper and sample *E* being within the specification requirements. Sample *C* shows:

Copper	0.08	per cent
Antimony	0.08	"
Tin	0.08	"
Zinc	0.08	"

Sample *F* is a standard showing 0.015 per cent zinc.

In Fig. 11 are shown spectrograms of four samples of lead, *A* being spectrographically pure, *B* a standard containing 0.01 per cent cadmium and 0.01 per cent nickel, *C* a sample from a shipment showing both cadmium and nickel in excess of specification, and *D* one meeting specifications.

Figure 12 shows four babbitt metals of which sample *A* is a standard containing:

Zinc	0.08 per cent
Iron	0.12 "
Aluminum	0.10 "

Sample *B* is within these limits, *C* shows 0.11 per cent zinc with iron and aluminum within requirements, while *D* shows 0.22 per cent zinc, with iron and aluminum within limits.

Without giving further illustrations of this particular nature, we may well claim that spectroscopy, properly used, will enable a control or testing laboratory to save a great deal of time and money in testing products when it is necessary to know whether a particular element or certain elements are present in less than a specified amount. It should be quite obvious, however, that the spectroscopist is limited to extremely low percentages, or fractions of 1 per cent in some cases, in the present state of our knowledge.

We are then faced with the problem of future developments of spectroscopic control analysis. Shall we be enabled to raise the limits of percentage determination by more careful plotting of lines appearing only at certain percentages, and possibly, though not probably, going into the relatively unexplored infra-red field? Or, and this seems the more likely, shall we follow the lead of the excellent work done by Prof. Hartley[1] and followed by the New Jersey Zinc Co. in a very special field, namely, make solutions of the materials under consideration, and when a certain line or certain lines are too intense for measurement or comparison, cut down the strength of the solution by taking aliquot portions and diluting until a line of known, or measurable, intensity appears?

With the knowledge that we have saved an immense amount of money, not only in the time of the laboratory force, but in reduction of inventory by prompt release of stock; with the knowledge of greatly accelerated testing methods; with the knowledge that we have solved many problems practically insoluble by wet or other customary methods of analysis; we have presented this short sketch of some of our work in the hope that criticism, friendly or otherwise, will be of benefit, not only to ourselves but to others.

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[1] "Wave Length Tables for Spectrum Analysis," by T. Twyman, Adam Hilger, Ltd., 75 Camend Road, London, N. W. 1.

CONCLUSION

The authors are firmly convinced of the great usefulness of the quartz spectrograph at present in the field of qualitative analysis. They are likewise of the opinion that a still greater field of usefulness lies in the direction of quantitative analysis, but that such development will come only after considerable collaboration between interested laboratories, and particularly with the production of accurate standards, without which quantitative examinations are hopeless.

DISCUSSION

Mr. Clamer. THE CHAIRMAN (*Past-President G. H. Clamer*[1]).—At a meeting of Committee B-2 on Non-Ferrous Metals and Alloys yesterday, the question was raised as to the interpretation of the word "none" as applying to the aluminum content in certain brass and bronze mixtures. The word "none" or "trace" is a very elastic term, subject to wide interpretation, and it was contended that it should be defined. The aluminum as determined by the A.S.T.M. method and reported as none, might still be quite high. Mr. Lundell, of the Bureau of Standards, has devised a more sensitive volumetric method, but I imagine this spectrographic method will be still more sensitive.

Mr. Hull, have you had any experience in the determination of aluminum in brass by this method as compared with the volumetric method?

Mr. Hull. MR. F. A. HULL.[2]—Once or twice we have had occasion to do that, and it was not particularly satisfactory because it was difficult to get a standard to go by. Spectrographically pure aluminum was not obtainable at that time, and consequently we could not make a proper determination. It is very difficult to get spectrographically pure elements, and outside of lead, zinc, cadmium, and copper, I think we have reached our limit at this time on spectrographically pure elements.

Mr. Davis. MR. C. H. DAVIS.[3]—The determination by means of the spectrograph of the presence of aluminum in an alloy is a simple matter. Quantities less than 0.01 per cent are readily detected. The same is true of many other elements, particularly the metals, such as copper, silver, tin and cadmium.

The accuracy of the spectrographic determinations of small quantities of aluminum and of other elements is often greater than those of wet analysis. The sensitivity of the test depends, however, on the nature of the element to be studied, and not to any marked degree on its state of combination in an alloy.

In the laboratory of The American Brass Co., systematic quantitative work has not as yet been done. The comparison of many spectrograms with the results of wet analyses has made it possible to estimate the relative amounts of the elements present, particularly when the percentages are small.

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[1] President and General Manager, The Ajax Metal Co., Philadelphia, Pa.

[2] Chief Chemist, Testing Laboratory, General Electric Co., Schenectady, N. Y.

[3] Technical Department, The American Brass Co., Waterbury, Conn.

Mr. Tree.

MR. R. M. TREE.[1]—It would be interesting to know how the elements used as standards were prepared in the spectroscopically pure state.

Mr. Hull.

MR. HULL.—I am sorry to say that I cannot answer that, because we got one from Mr. Cowan, of the National Lead Co., and the others from the New Jersey Zinc Co. If there are representatives of those companies here, they may be prepared to tell you how they made them. I cannot tell you.

Mr. Finkeldey.

MR. W. H. FINKELDEY.[2]—The pure metals, zinc and cadmium, are prepared by several distillations at relatively low temperatures in a vacuum, starting with the purest metals obtainable commercially. The process is really one of fractional distillation, since only the purest section of the condensed zinc is used for further treatment; the residue of undistilled metal and the section of condensed zinc adjacent to the deposit of the more volatile impurities is discarded after each distillation. This procedure is repeated until spectroscopic examination of the metal shows the absence of all lines of any other metallic element except the metal being purified. Usually three such distillations are required.

Mr. F. G. Smith.

MR. F. G. SMITH[3] (*by letter*).—I have been particularly interested in the possibilities of spectrum analyses, inasmuch as I did a large part of the pioneer work with the spectroscope which was used by Bassett and Davis in the preparation of their paper which is referred to. I wish to submit the following suggestions as discussion of the paper in the hope that they will be of value to the authors.

The work of A. de Gramont on spectrum analysis is sufficient evidence that quantitative results can be obtained with the spectrograph. I do not know whether the method he suggests has ever been more fully investigated, but it apparently has possibilities. A. de Gramont shows, when the source of energy used for the spark between metal electrodes is a fairly high-tension alternating current, that with a fixed capacity in circuit it is possible, by adding inductance, to eliminate all the air lines from the spectrum. If the inductance is further increased, many spectrum lines from the metal electrode will also disappear until finally only a few remain. These latter de Gramont has called the *raies ultimes*. Knowing these facts, is it not reasonable to assume that the character of the light emission from metal electrodes is a function of the alternating current circuit conditions which may affect the frequency of oscillation of the electrical discharge?

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[1] Technical Supervisor, The American Brass Co., Kenosha, Wis.

[2] Assistant Chief of Research, Research Division, New Jersey Zinc Co., Palmerton, Pa.

[3] Metallurgist, The American Brass Co., Waterbury, Conn.

Mr. F. G. Smith. My ideas are these: Assume that one wishes to determine an impurity in a metal, such as bismuth in lead. If the lead is used as electrodes with a fixed source of alternating current energy, there will be a definite combination of inductance and capacity which will suppress all or most of the lines of bismuth depending upon the quantity; or, there will be discovered one or more lines which show a great variation in intensity due to changes in the circuit. Next, if one has a number of samples of lead with varying amounts of bismuth, the amount or combination of inductance and capacity will vary, if in the spectogram of each sample all but certain lines of bismuth are suppressed, or the intensity of one or more lines is kept constant. Finally, if this assumption is correct, it then becomes necessary, in order to make quantitative determinations of bismuth in lead, to calibrate the combinations of inductance and capacity required to produce standard spectrum negatives of the impurity, from standard samples of lead in which the impurity is known. For example, in Fig. 8 of Hull and Steele's paper there are shown spectrograms of seven samples of lead in which the bismuth lines vary in intensity. With conditions under which these were photographed let us assume sample G with 0.05 per cent of bismuth and its spectrogram as the standard. The bismuth line at the right is only faintly seen in the spectrogram. If this line (or some other more sensitive line) in all the other spectrograms were brought to the same intensity under standard photographic conditions, by means of changes in the inductance and capacity in the circuit, then those changes would be a quantitative measure of the amounts of bismuth and would constitute the calibration.

I would suggest that the following experiment be tried with a sample of lead carrying an unknown small amount of bismuth. First make the standard exposure of the standard sample G for comparison. Then make a series of standard exposures of the unknown, varying the circuit in steps as above indicated. The circuit conditions which produce a line intensity like that of the standard G will indicate the amount of bismuth from their calibrated values.

This appears to be more devious than the method of direct comparison with standard spectrograms, but I believe that if this method can be worked out it would be possible, after a little experience, to take a series of standard exposures on an unknown sample and to recognize changes in the spectrum due to definite changes in circuit conditions, which would indicate quite accurately the amounts of impurities without a direct comparison with a standard spectrogram, or a series of standard spectrograms from standard samples. In

Mr. F. G. Smith.

other words an "end-point spectrogram" of small amounts of impurities might be obtained by making definite changes in the circuit. The significance of the changes might be calibrated as suggested.

Mr. Meggers.

MR. W. F. MEGGERS[1] (*by letter*).—We are tremendously interested in this very excellent paper. During the past twelve years we have regularly been using our spectrographs for qualitative and for quantitative analyses, largely of materials for which the ordinary chemical methods are slow or inadequate. We have been recommending the method to the industries and we are delighted to see that they are coming to recognize the usefulness of a spectrograph in their laboratories. We consider that the paper by Hull and Steele contains many well-chosen illustrations of the economies which may result from the practical use of a spectrograph.

Mr. D. M. Smith.

MR. D. M. SMITH[2] (*by letter*).—The conclusion of the authors with regard to quantitative spectrum analysis, that without the production of accurate standards "quantitative examinations are hopeless," has a discouraging sound. It should be remembered that in a metallurgical laboratory there are always many accurately analyzed samples containing varying percentages of some impurity which is of interest. These provide the necessary accurate standards on which reliable quantitative examination of further samples can be based.

Cadmium, Lead and Iron in Zinc.—We have carried out a research on the spectrographic determination of small quantities of impurities in zinc for the British Non-Ferrous Metals Research Association, who supplied very carefully prepared samples of zinc and graded alloys for standards, including one sample from the New Jersey Zinc Co., prepared by fractional distillation after electrolytic refining and considered to be spectroscopically pure.

Routine methods were developed for the following impurities (details will appear in the *Transactions* of the American Institute of Mining and Metallurgical Engineers):

Cadmium.................	up to 0.75 per cent Cd
Lead.....................	up to 0.1 per cent Pb
Iron.....................	up to 0.1 per cent Fe

The method adopted was to photograph either the arc or spark spectra of the samples. Both arc and spark are suitable for the determination of cadmium, but for the determination of lead the arc, and for iron the spark is more suitable.

The method of comparing intensities proposed by W. Gerlach is very well illustrated by the determination of cadmium in zinc by

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[1] Chief, Spectroscopy Section, U. S. Bureau of Standards, Washington, D. C.

[2] Research Laboratory, Adam Hilger, Ltd., London, England.

Mr. D. M. Smith. means of spark spectra. The ordinary condensed spark (without the inclusion of a self-induction coil) is used in this case.

The two cadmium lines λ2144.4 Å and λ2573.0 Å are chosen, since from the appearance of these lines alone the estimation can be carried out. The line λ2573.0 Å appears faintly between two zinc lines when there is 0.25 per cent of cadmium present, and is of approximately the same intensity as the two zinc lines when 0.75 per cent of cadmium is present. The relative intensities of the cadmium line λ2144.4 Å and the neighboring zinc line λ2147.4 Å enable one to distinguish clearly between the presence of 0.001, 0.01 and 0.1 per cent of cadmium.

Bismuth in Copper.—Another example may be quoted, namely, that of bismuth present as an impurity in copper. When there is 0.1 per cent of bismuth in copper, the arc spectrum of the metallic sample shows the bismuth line λ3067.7 Å approximately equal in intensity to the copper line λ3073.8 Å.

Analysis by Visual Spectrometry.—There are many instances where a visual examination is to be preferred, either on account of the much shorter time involved in the analysis, the evanescence of the traces sought, or because the materials to be examined cannot be made the electrodes for an arc or spark.

There are but few substances containing metals which cannot be spectroscopically examined in a carbon arc. The technique is simple and readily carried out and has been constantly used by Herbert Jackson (Director of the British Scientific Instrument Research Association) for many years.

It is an advantage to use a cored carbon for both electrodes, the most suitable carbons being about 15 cm. long and 1 cm. in external diameter, the diameter of the core being about 2 mm. They should be purified by successive prolonged soakings in hydrochloric acid after which they are soaked and washed in distilled water. After being dried they are ready for use.

A blank test is made by running the arc for a few minutes to make sure that the sensitive lines which are to be employed are not visible as a constituent of the purified carbons. Before making the tests, the core of the positive carbon is removed with a small drill to a depth of about $\frac{1}{4}$ in. The current having been turned off and the positive carbon having cooled sufficiently, the material under examination is introduced into the cavity of the positive carbon. The arc is again struck and the spectrum examined for the sensitive lines with a suitable visual spectrometer, which must have sufficient dispersion.

Mr. D. M. Smith.

Where very minute amounts of impurity are suspected and where chemical processes of separation are inapplicable, it may be advisable or necessary to concentrate the impurity. For example, in testing for minute traces of copper in zinc oxide it is a simple matter to increase the percentage of copper in a portion to be examined spectroscopically by precipitating the copper along with some zinc. It is, as a rule, unnecessary to attempt to remove the whole of the main constituent and the tedious process of complete chemical separation is avoided.

Messrs. Hull and Steele.

MESSRS. F. A. HULL and G. J. STEELE[1] (*authors' closure by letter*).—The contribution to the discussion by Mr. F. G. Smith of Waterbury, Conn., has had our due consideration and we agree with him that the proposition as submitted is one worth while investigation. We regret that we have not carried out the suggestion offered as our present equipment is not capable of handling the situation.

The expense incurred in additional apparatus would be costly and inasmuch as this investigation may have had the attention of others, some established facts may be brought to light.

Should the suggestion develop any real merits, we are afraid that in practice, where several elements have to be estimated in the same sample, it would necessitate the taking of too many spectrograms.

With reference to the preparation of standards used in quantitative analysis and our statement that without them, successful work would be hopeless, referred to by Mr. D. M. Smith of Adam Hilger, Ltd., we had in mind the fact that in the estimation of impurities ranging from 0.003 to 0.15 per cent no two laboratories might agree.

This same situation has been taken care of in other ways by the Bureau of Standards, who in arriving at its final results takes the average of not less than ten industrial analyses. This laboratory has been a cooperator in such standardizations.

We are particularly interested in Mr. Smith's research on the impurities in zinc, with special reference to the estimation of cadmium up to 0.75 per cent by the Gerlach system of comparing intensities.

The example is very well put and should serve as an outstanding case, illustrating the fact that the question of unequal exposure can be in some cases combatted by the Gerlach system.

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[1] Testing Laboratory, General Electric Co., Schenectady, N. Y.

A RAPID AND PRACTICAL METHOD OF APPLYING THE FERROXYL TEST TO PROTECTIVE COATINGS

By Karl Pitschner[1]

Synopsis

The "ferroxyl" test for determining the presence of pinholes in plated coatings has been in use for a long time but up to the present has been a laboratory test rather than a shop test, due to difficulties in the application of the reagent and in obtaining a permanent record. A search for a more practical mode of handling, led the author to the development of a test paper embodying the reagent by means of which it is possible to make a permanent record in two or three minutes. The reagent has been developed so that it will detect pinholes through nickel or chromium to copper as indicated by brownish red spots.

The advantages of this method of application are enumerated, and comparison of the test paper records with salt spray results are shown.

The principle of the "ferroxyl" reagent has long been used in the detection of porosity in electroplated coatings on ferrous metals. The usual method of applying the test has been described by Wm. Blum and C. F. Thomas in a paper presented in September, 1925, before the American Electrochemical Society.[2] Briefly, a hot solution containing potassium ferricyanide ($K_3Fe(CN)_6$), sodium chloride ($NaCl$), and agar-agar is flowed in a thin layer over the surface to be tested. This solution quickly hardens to a jelly as it cools. The sodium chloride reacts with the iron exposed at the pin holes. A little iron goes into solution and reacts with the potassium ferricyanide, forming a deep blue coloration in the immediate vicinity of the hole in the coating which is probably Prussian blue, a mixture of a number of ferrocyanides. If a permanent record is required, the layer of jelly is removed intact to glass or paper and dried.

In practice, this method of applying the "ferroxyl" reagent requires careful manipulation and proves very tedious. The operation must be performed in a dust-free atmosphere to prevent contamination of the solution. This fact excludes its use as a practical plating room test. The reagent must be kept hot in a water bath which is usually not convenient. With continued heating it is almost impossible to keep the solution from becoming lumpy, due to a film drying on the surface and later breaking up into small bits. If a permanent record is desired, the solution must contain exactly the right concentration of agar-agar, so that it will be sufficiently fluid to flow readily over the surface and still harden quickly to the point where it may be removed intact. The author found, at the beginning of his work on this reagent, that substitution of 20 per cent of ethyl alcohol for that amount of water improved both flowing and hardening qualities. In making a permanent record, the transference of the jelly film from the surface tested to paper requires the most careful handling, and even then very frequently a breaking of the film results. Necessarily, the surface from which a permanent record can be made is very small, at the most eight to ten square inches. In drying the film on paper, the agar shrinks, the paper is discolored and warped, the original dimensions of the area tested are lost, and the salt frequently crystallizes out, which results in an unsightly and unreliable record.

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[1] Chief Chemist, Research Department, American Chain Co., Bridgeport, Conn.

[2] *Transactions*, Am. Electrochemical Soc., Vol. XLVIII, p. 69 (1925).

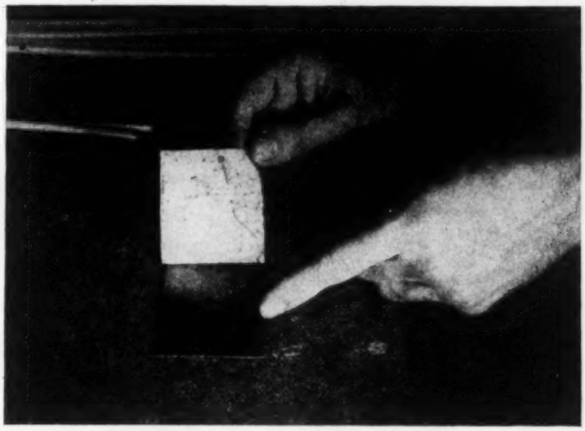


FIG. 1.—Showing Method of Making Ferroxyl Test by Means of Prepared Paper.

These difficulties and faults in the application of the "ferroxyl" test as a practical routine method for platers led the author to study various means of improving this test and adapting it to use in the shop as well as the laboratory. The idea was conceived of coating paper with a thin film of the reagent and immediately spreading it over the surface to be tested, allowing it to remain for a few minutes. Fairly good records were obtained in this way. Later it was found that the reagent could be sprayed on the paper, dried in an oven and the paper kept indefinitely without deterioration. Paper prepared by this second method was merely moistened on the sensitized side and spread over the dry surface to be tested. This method of application is shown in Fig. 1. After removal it was found possible to wash all the reagent out of the paper with water, leaving only the blue spots indicating the pin holes.

The first prints made with the sensitized paper showed the pin holes only with a light blue color. The "ferroxyl" reagent for treating the paper had been made up according to directions in the paper by Blum and Thomas, mentioned previously. While this solution was found very satisfactory when spread in a comparatively thick layer directly on the surface to be tested, it was evident that the concentration of the compounds entering into the reaction was not sufficiently high to get the best results with a thin film of the dried reagent on paper. Accordingly, the concentration of the various ingredients was increased 50 to 100 per cent. A part of the water was replaced by ethyl alcohol. This altered solution was filtered while hot through glass wool in a 250-cc. dispensing burette held over a gas hot-plate to keep the solution from solidifying. This filtration removed all foreign material and clots of blue coloration in the solution.

The paper sprayed with the altered reagent proved very satisfactory. The blue coloring at the pin holes became intense. The boundaries of the spots became very definite due, no doubt, to greater amount of agar-agar present. At this time the paper was being tested on a double coating of nickel plate on copper plate on steel. A few brownish-red spots were noticed on some of the test papers which had been used. Tests on a nickel plate on pure copper sheet proved that the red spots were due to pin holes through to copper but not to steel. The brownish-red compound formed is probably copper ferrocyanide. It was later found that by using a solution of ammonium citrate and ammonium carbonate instead of water for moistening the paper, a record could be obtained of the porosity of nickel or chromium plate on a copper or brass base which was fully as definite as the print through to ferrous base. However, the solution of ammonium citrate should not be used when porosity through to a ferrous base is to be tested, as it hinders the formation of the blue coloration. For testing automobile bumper bars, first plated with copper and then with nickel, the test paper will show pin holes through to the iron, pin holes through to the copper, and the points where the nickel is almost buffed through to the copper.

We have found that this altered method of applying the "ferroxyl" test to determine porosity has the following advantages:

1. No equipment is required.
2. The only supplies needed are test paper and water. If the material to be tested is greasy, a solvent like carbon tetrachloride should be used to clean the surface before applying the paper.

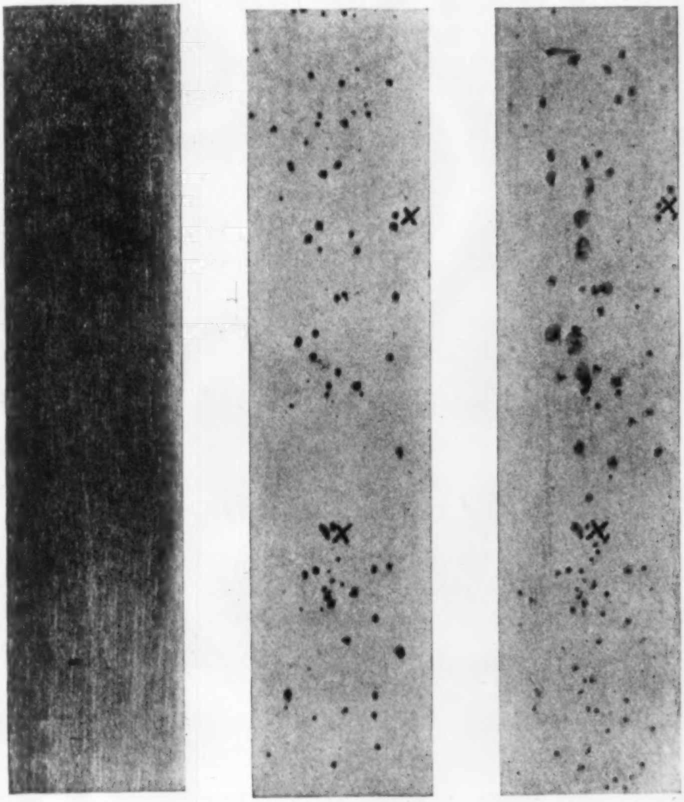
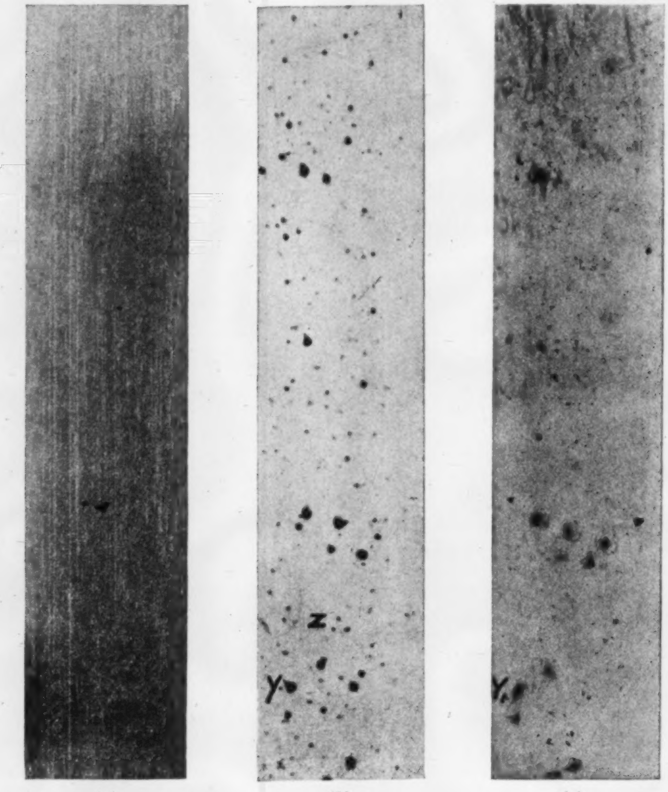

(a) (b) (c)

FIG. 2.—Application to Nickel-Coated Surface.

3. Contamination from dust in the air is reduced to a minimum by the rapidity and mode of application.
4. Any person can apply the test without previous instruction.
5. The test is complete in about three minutes including washing out of excess reagent.
6. The test is non-destructive. If the bar is washed off soon after the paper is removed, buffed surfaces are not even tarnished.

7. A permanent record of quality of production in the plating plant is obtained. All of the above features make this method of applying the ferroxyl test of value to plating departments and to buyers of electroplated work and tin plate.

8. Any size prints may be obtained by laying down adjoining sheets of the paper. The sheet used is, of course, limited to a size and shape which can be rapidly and conveniently handled.



(a) (b) (c)

FIG. 3.—Application to Nickel Over Copper-Coated Surface.

9. The porosity of material such as tin and aluminum foil may be tested by placing the foil on a surface of iron and applying the paper on top of the foil.

10. For testing nickel and chromium plate, the ferroxyl test as compared with the salt spray will show up in three minutes all of the failures which the salt spray will develop in 25 to 50 hours. If a print is made by means of the paper and the sample then put into the salt

spray, the points of failure of the coating can be checked point for point by comparing the print and the bar from salt spray.

Figures 2 and 3 are intended to illustrate the relation between the spots on the paper and the corrosion of the steel beneath the plating.

Figure 2(*a*) shows a piece of steel bumper bar plated with about 0.0007 in. of nickel, direct on the steel. The ferroxyl print made at the time that the original bar was photographed is shown in Fig. 2 (*b*). All of the spots in this case are dark blue, showing pin holes through the nickel plate to the ferrous base. Figure 2(*c*) shows the bumper bar after 45 hours in the salt spray. A comparison of Figs. 2(*b*) and (*c*) shows that practically all points of failure under the salt spray were predicted beforehand by the spots on the ferroxyl paper. Points marked X and X_1, for example, show the same groups on the test paper and on the steel bar after the salt spray.

Figure 3 shows the same prints made of another bar which was first plated with copper, then with nickel. A close observation of Fig. 3(*b*) will disclose two classes of spots of different intensities. The dark spots, comparatively few in number, show pin holes through both plated coatings to the ferrous base metal. These were colored a deep blue on the original print. A typical spot of this type is shown at Y on Fig. 3(*b*) of the test paper print. Y_1 on Fig. 3(*c*) shows the same locality as a rust spot on the steel bar after 45 hours in the salt spray. The many lighter spots typically shown at Z in Fig. 3(*b*) are colored a reddish brown on the test paper, indicating a pin hole through the nickel to the copper plate, but not extending through the copper to the steel. A comparison of Figs. 3(*b*) and (*c*) shows that this class of spots does not appear as rust spots on the bar when removed from the salt spray after a period of 45 hours, due to the protection of the copper.

DISCUSSION

Mr. Hogaboom. MR. GEORGE B. HOGABOOM[1] (*presented in written form*).—This method of testing nickel deposits is most opportune. The demand for good nickel plating, one that will afford a better protection against corrosion, is insistent and is a necessity especially in the automotive industries. It is well known that nickel does not protect steel or iron against corrosion in the same manner as zinc. Rather it accelerates corrosion. The only condition under which a nickel deposit can be of any protection is for the nickel to be free from porosity.

The salt-spray test for determining or computing the life of a nickel deposit is not satisfactory. This test has never been standardized, yet it is universally applied to nearly all electroplated deposits and in many specifications a definite duration of a deposit in a salt spray is required. It was developed for testing zinc coatings, and without any data as to its value has been designated as a test for nickel deposits.

Zinc has the property of exerting a protective influence over any small uncoated surface. The value of the salt spray test lies in the rate of solubility of zinc in a salt solution. It detects thin deposits more rapidly than it does actual porosity. Nickel does not have the same rate of solubility in a salt solution as zinc. It does not have that protective influence over any uncoated surface. The value of the salt spray test is based upon its attack of the base metal which is exposed due to the porosity of the deposit. The exposed part is often so small that the created rust is washed off by the spray and cannot be detected. While rough comparative tests can be made they are in no way reliable, and the data obtained from a series of tests at one manufacturing plant are not comparable with tests made at another plant. At its best the salt spray test does not give the accuracy desired. At a recent meeting of the Electroplaters and Depositors Technical Society, a branch of the Faraday Society in England, this was recognized and a resolution was passed calling for the standardization of the salt spray test.

The value of the ferri-cyanide, and agar-agar method is given in the paper under discussion.

The Pitschner paper describes a unique and reliable method for obtaining permanent and comparable data on the porosity of nickel deposits. The method is simple and rapid and furnishes a record which makes it ideal. This test paper described has also been found of assistance in detecting thin deposits of nickel from solutions having poor throwing power, that is, the ability to deposit metal in deep recesses. It also indicates uneven distribution of deposits due to the incorrect placing of anodes or to not replacing worn anodes. From solutions that have poor throwing power, the deposit in deep recesses is thin and consequently very porous. By testing the deposition on the high lights and on the recesses with Pitschner's paper a rapid method of determining the throwing power of a nickel solution is had. While this is by no means accurate it is a good indication and is convincing to a person not versed in technical methods. If a piece of work is plated directly in front of an "old" anode, one that is almost entirely corroded, and another piece of work in front of a "new" anode the former will have considerably less nickel deposited on it. This is readily shown by the porosity of the thin deposit brought out by Pitschner's paper and offers a visible proof to those in charge of the electroplating. This point clearly demonstrates the value of a moving cathode arrangement where all work travels in front of every anode, regardless of its area, the same length of time. In this way alone can a uniform deposit be obtained. **Mr. Hogaboom.**

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[1] Research Electroplater, Hanson and Van Winkle Co., Newark, N. J.

Mr. Pitschner is to be commended for bringing out this clever and simple method for testing the porosity of nickel deposits. It is a test that is far more reliable than any that has previously been developed.

Mr. Flowers. MR. A. E. FLOWERS[1].—The authors are to be congratulated on preparing a method of testing which also gives a permanent record. That has been one of the principal disadvantages of so-called gelatine methods. A minor difficulty with the gelatine methods has been the fact that after the coating has been applied, if you wish to preserve it for any time, there is a tendency for it to crack and curl off. We have found that we could eliminate most of that difficulty by the addition of a small amount of glycerin. But this leaves the coat slightly tacky to the touch, which may or may not be a disadvantage. We, however, apply these methods rather infrequently, placing most of our dependence on a fairly simple test which consists merely of an open-work wood rack about a foot above the floor with small jets of steam from pet cocks below. The steam rises and condenses on the under surface of the pieces laid upon the test rack. A 24-hour exposure to such a test has, in every case we know of, shown up the pinholes or uncovered spots on hot tin pieces.

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[1] Engineer, In Charge of Development, The De Laval Separator Co., Poughkeepsie, N. Y.

Mr. Blum.

MR. WILLIAM BLUM[1] (*by letter*).—This paper is of special interest because recent observations indicate that for metal coatings such as nickel, copper and chromium on steel the porosity furnishes a definite indication of the probable protective value. This simple method of testing and recording the porosity should therefore prove useful in the inspection of products plated with such metals.

Even though the exact concentrations of the various constituents may not be significant it would be desirable for purposes of record and comparison for the author to state in the paper the exact concentration of each of the constituents which he has found most convenient.

At the bottom of page 306 reference is made to the detection of "points where the nickel is almost buffed through to the copper." This statement should be amplified, especially to explain whether such behavior indicates that the nickel coating, however thin, is itself appreciably attacked by the reagent in the test paper.

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[1] Chemist, U. S. Bureau of Standards, Washington, D. C.

THE TENSILE STRENGTH OF PORTLAND-CEMENT CONSTITUENTS

By Jasper O. Draffin[1]

Synopsis

The paper presents a method of estimating the strength of portland cement at various ages from a study of the variation in the strengths of its three principal constituents.

Data on the strength of the three constituents, prepared from pure materials and tested in tension, have been taken from publications of the U. S. Bureau of Standards and used to estimate the strength of an average portland cement. Comparison is made between the computed and the actual strength. The computed strength is lower than the actual strength, but the shape of the age-strength curve agrees with that of one of the curves determined by experiment.

This method of estimating the strength of cement is not practicable as a general method but is suggestive of future possibilities. The need for more related data is emphasized.

The strength of portland cement cannot be measured by the extent to which a single property or quality is present; the tensile strength, compressive strength, resistance to abrasion, adhesion to aggregate, resistance to solution by water, permeability and durability are all properties of importance in the varied uses of cement, and information concerning each of these properties is essential to a complete knowledge of the material. Nevertheless, it is usually necessary to study each property separately and then attempt to evaluate the material on the basis of part or all, as the case may be, of the factors. This paper presents the results of a study of the tensile strength of portland cement using as basic information a series of tests made at the U. S. Bureau of Standards.[2] In these tests the more important compounds in portland cement were prepared from pure materials and mixed with each other in definite proportions. These mixtures were then made into neat cement and sand-mortar briquets and tested in tension. The results of these tests are analyzed and presented herewith.

Strength of Portland Cement Constituents

It is fairly well agreed[3] that the principal constituents in portland cement are tri-calcium aluminate ($3CaO \cdot Al_2O_3$), tri-calcium silicate ($3CaO \cdot SiO_2$), and Beta di-calcium silicate ($\beta\ 2CaO \cdot SiO_2$). It is also generally supposed that the 24-hour strength of cement is due chiefly to the tri-calcium aluminate; the 30-day strength to the tri-calcium aluminate and the tri-calcium silicate, and the increase in strength after this period to the di-calcium silicate.

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[1] Assistant Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] Bates and Klein, "Properties of the Calcium Silicates and Calcium Aluminate Occurring in Normal Portland Cement," U. S. Bureau of Standards, *Technologic Paper No. 78*.

[3] R. H. Bogue, *Concrete, Cement Mill Edition*, January, 1927, p. 33.

The proportions of the different constituents which were used in the Bureau of Standards tests are shown in Table I. Each mixture is represented by the percentages shown in a horizontal line in the table and was prepared by taking the indicated percentage of each pure constituent and grinding the constituents together. In a parallel series, some, but not all of the mixtures were also prepared by burning the mixtures and grinding the resultant clinker. All mixtures were prepared both with and without the addition of gypsum. The resultant material, over 80 per cent of which passed a 200-mesh sieve in all but two cases, was used in the preparation of neat-cement and 1:3 sand-mortar briquets, which were tested in tension at various ages.

TABLE I.—PERCENTAGES OF THE THREE CONSTITUENTS OF PORTLAND CEMENT WHICH WERE PREPARED, MIXED TOGETHER, AND GROUND.

TRI-CALCIUM ALUMINATE	TRI-CALCIUM SILICATE	DI-CALCIUM SILICATE
100	..	..
..	100	..
..	..	100
19	81	..
19	..	81
..	50	50
19	40.5	40.5

The strength-contributing value of each constituent has been determined by first considering the strength of the briquets made of pure tri-calcium aluminate, tri-calcium silicate, and di-calcium silicate, which gave the strength of each constituent in a pure state. The strength of any mixture of these constituents might be expected to be the sum of the strengths of the separate constituents. This composite strength was, however, modified by the restraining or accelerating action of each constituent in the mixture on the others present, so that the strength of the mixture was always greater than the sum of the strengths contributed by each compound. This increase in strength was apportioned among the constituents in proportion to the amount which each was first assumed to contribute to the strength of the mixture. By computing the strength of the various mixtures separately and by taking averages of all the combinations tested, the values have been determined from which the curves shown in Fig. 1 are plotted. The values were determined in terms of the tensile strength, in pounds per square inch, contributed by each one per cent of the constituent.

Stated differently, the curves show the unit strength which each one per cent of constituent contributes to the strength of the mixture. Suppose, for instance, that a cement is made up of 20 per cent of tri-calcium aluminate, 40 per cent of tri-calcium silicate and 40 per cent of di-calcium silicate. The unit strength of these at 6 months is respectively 1.83, 7.74 and 7.16 lb. per sq. in. The total strength of the neat cement at 6 months will then be 20 × 1.83, plus 40 × 7.74, plus 40 × 7.16, a total of 633 lb. per sq. in.

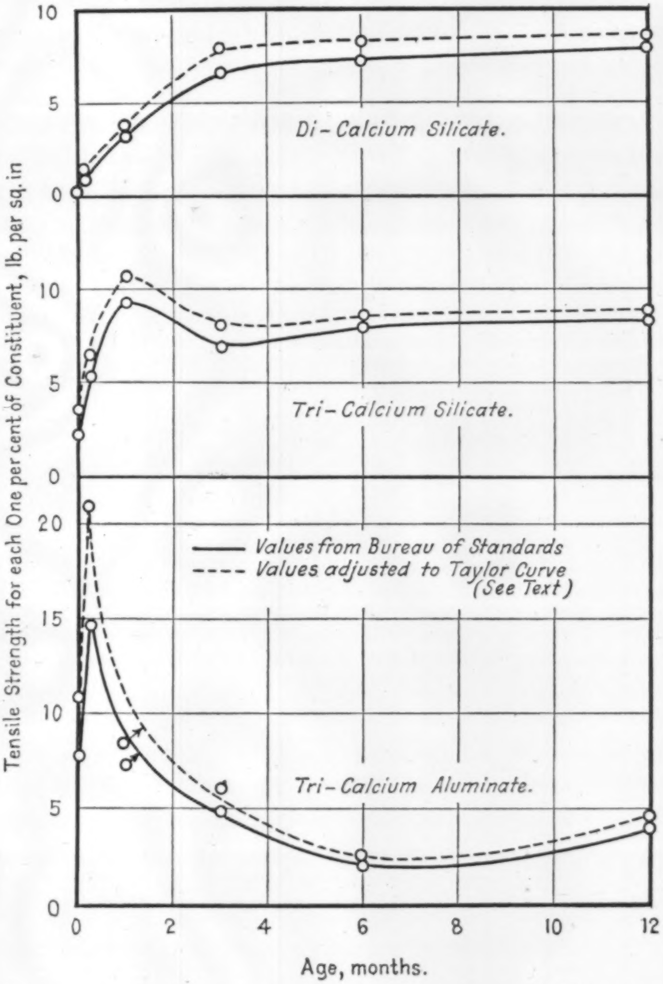

FIG. 1.—Curves Showing the Tensile Unit Strength at Various Ages of Three Major Constituents in Portland Cement.

The tests made on 1:3 sand-mortar briquets were analyzed in the same manner as were those of the neat cement and the values are plotted in Fig. 2. The values are of course lower than those for neat cement, and since there were not as many specimens of the sand mortar tested as there were of neat cement, the data are not as consistent as are those for the neat cement, especially at 3 months and 6 months.

STRENGTH OF PORTLAND CEMENT AND MORTAR

Determination of Amount of Each Constituent in Cement.—In order to use these curves it is necessary to determine the amount of each of the three constituents in cement. The method of computation used is similar to that used by geologists in computing the percentage of minerals theoretically present in an igneous rock of any known chemical composition[1] and has been used for computing the percentage of the constituents of portland cement.[2] The procedure is as follows: The alumina, silica and lime are considered and the percentage of each of these is converted into molecular proportions by dividing the percentage of each oxide by its molecular weight as illustrated in the second and third columns in Table II. The alumina is first assigned its proper amount of lime to form tri-calcium aluminate and the remaining lime is then apportioned to the silica to form tri-calcium silicate and di-calcium silicate in such amounts as will utilize the lime and silica. The molecular proportion of each of the resulting compounds is multiplied by the molecular weight of the compound in order to obtain the percentage of the compound, as shown in Table III. Since the sum of these compounds does not equal 100 per cent, due to the omission of the iron, magnesium and other oxides, these percentages are adjusted to 100 per cent.

TABLE II.

Oxides	Percentage	Molecular Proportion	Tri-Calcium Aluminate	Tri-Calcium Silicate	Di-Calcium Silicate
Silica	21.47	0.358	.....	0.180	0.178
Alumina	7.27	0.071	0.071	.....	.....
Lime	62.01	1.110	0.214	0.540	0.356

The magnesium oxide apparently does not enter into a chemical combination but exists as a solid solution[3]; the iron oxide may form two compounds, mono-calcium ferrite and di-calcium ferrite,[4] but these have been observed in portland cement in only very small quantities.[1,2] For these reasons the iron and magnesium compounds are neglected in computing the amount of the constituents of portland cement. This does not imply that they have no strength but simply that their strength properties are unknown.

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[1] J. R. Iddings, "Igneous Rocks," Vol. I, 2nd Ed., pp. 435–444 (1920).

[2] R. J. Colony, Am. Inst. Mining and Metallurgical Eng., Vol. LXVI, pp. 66–81 (1922).

[3] G. A. Rankin, "The Constituents of Portland Cement Clinker," *Journal*, Industrial and Engineering Chemistry, Vol. 7, No. 6, p. 466 (1915).

[4] Sosman and Merwin, *Journal*, Washington Academy of Science, Vol. 6, p. 532 (1916).

Comparison of Estimated and Actual Strength of Cement and Mortar.—If the tensile strength of portland cement and mortar is governed by the strength-giving properties of its various constituents and the curves in Figs. 1 and 2 represent the strength of these constituents in neat and sand mortars, the strength of cements as obtained by these curves and the actual strength of cements as found by tests should show some correspondence. Such a comparison is made in Fig. 3 between a computed age-strength curve for a typical cement and three actual age-strength curves. The computed curve is based on an "average" American portland cement which it is assumed may be represented by an average of seven analyses selected from a considerable number reported by Eckel.[3] These averages, expressed in percentages, are: silica 21.47, alumina 7.27, iron oxide 3.26, lime 62.01, and magnesium oxide 1.82. The percentages of the three constituents in this "average" cement, computed according to the method just outlined, are tri-calcium aluminate 21.2 per cent, tri-calcium silicate 45.1 per cent, and di-calcium silicate 33.7 per cent. The strength of the cement may now be computed by multiplying the percentage of each constituent by the unit strength of that constituent as taken from the curve in Fig. 1 and adding these values. The strength at different ages of this "average" cement is plotted in Fig. 3 with the Taylor,[4] Lesley,[5] and U. S. Geological Survey[6] curves. It will be

TABLE III.

Constituent	Molecular Proportion × Molecular Weight	Percentage Constituent	Adjusted Percentage Constituent
Tri-Calcium Aluminate	0.071 × 270 =	19.2	21.2
Tri-Calcium Silicate	0.180 × 228 =	41.0	45.1
Di-Calcium Silicate	0.178 × 172 =	30.6	33.7
Total		90.8	100.0

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[1] Hansen and Bogue, "Studies on the System Calcium Oxide-Ferric Oxide-Silica," *Paper No. 1*, Portland Cement Assn. Fellowship, May, 1926, p. 6.

[2] Klein and Phillips, "Hydration of Portland Cement," U. S. Bureau of Standards, *Tech. Paper No. 43*, p. 63 (1914).

[3] E. C. Eckel, "Cements, Limes and Plasters," 2nd Ed., pp. 523–524, 1922.

[4] Taylor and Thompson, "Concrete-Plain and Reinforced," 2nd Ed., p. 99 (1912).

[5] R. W. Lesley, *Journal*, Assn. of Engineering Societies, Vol. 15, p. 204 (1895).

[6] Humphrey and Jordan, "Portland Cement Mortars and Their Constituent Materials," Structural Materials Testing Laboratories, St. Louis, 1905–1907, U. S. Geological Survey, *Bulletin No. 331*.

noted that the shape of the computed curve is very like that of the Taylor curve, but the computed curve strengths are smaller. This may be due to a number of things: the strength of the iron and magnesium compounds is not included in the computed strength but these substances probably contribute appreciably to the strength of the cement; the iron and magnesium compounds may affect the action of the other compounds by acting as catalyzing or retarding agents and so increase the strength of the aluminate and silicate compounds above the values shown in Fig. 1; the storage conditions may not have been the same for different tests.

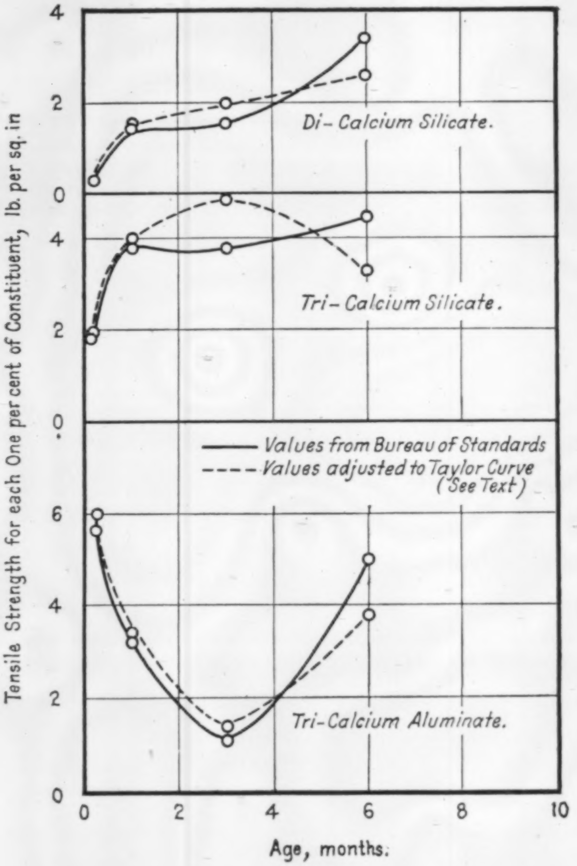

FIG. 2.—Curves Showing the Tensile Unit Strength at Various Ages of the Three Major Constituents in Portland Cement. 1:3 Sand Mortar.

Since the Bureau of Standards unit-strength curves give a strength for cement which is below the actual strength and since some of the strength-giving elements cannot be considered separately, it is obvious that the curves should be adjusted so as to include the additional strength elements which are present in all commercial cements. Accordingly, the Bureau of Standards curves have been adjusted so as to give the unit-strength of the three constituents as they exist in ordinary portland cement. This was done by apportioning the difference between the Taylor curve and the computed curve among the three constituents in proportion to the amount which they were originally assumed to contribute to the mixture and adding these amounts

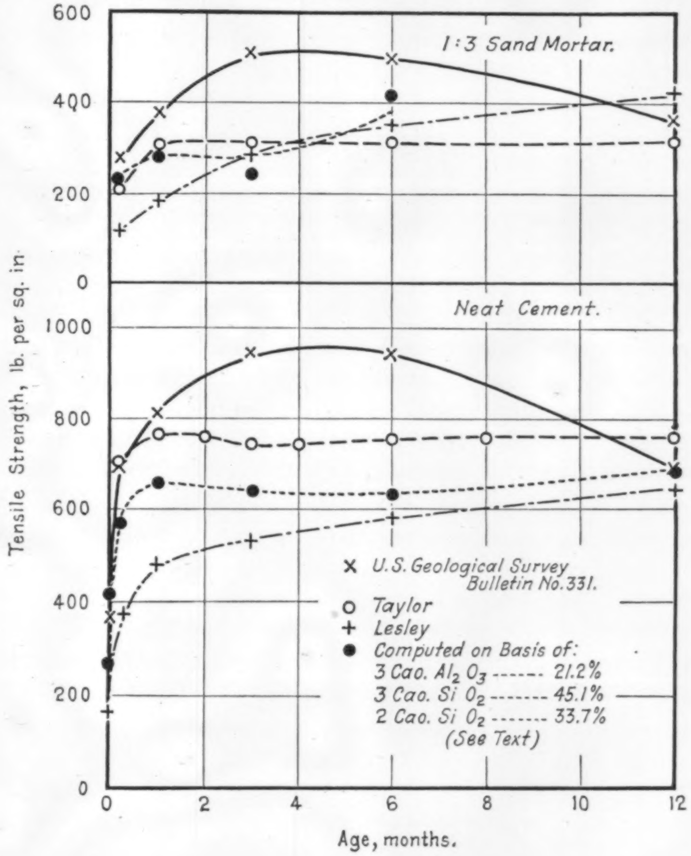

FIG. 3.—Comparison Between Actual and Computed Strength of Neat-Cement and 1:3 Sand-Mortar Briquets. Computed Curves Based on Data from Figs. 1 and 2.

to the Bureau of Standards values. The adjusted curve for each constituent has the same general form that it had before adjustment.

The computed and actual strengths of the sand mortars have been compared with each other in the same manner as were the strengths of the neat cements. The unit strengths are plotted in Fig. 2, and the strengths for the mortar made with the "average" cement, estimated from the unit strengths, are plotted in Fig. 3 together with the values taken from the Taylor curve for 1:3 sand mortar.[1] The unit-strength values for each constituent adjusted to the Taylor curve are also plotted in Fig. 2.

DISCUSSION

The supposition concerning the strength contributed by the constituents at different ages is seen to be reasonably correct.

An examination of the three typical curves for neat cement and sand mortar shows a marked difference in the general shape even though the chemical composition should be approximately the same. The Taylor curve is determined from a large number of tests and probably more nearly than either of the others is representative of the average composition which was assumed. It is suggestive that this curve, representing a large number of tests, agrees so well in shape with the computed curve. But, the fact that three series of tests sufficiently large in number that they may be assumed to be of approximately the same average chemical composition and, therefore, of the same constituent composition should have curves which differ so greatly in shape, shows the difficulty of obtaining a single series of values from which the strength of any given cement may be predicted. Surely, the variation in composition is not so great as to be responsible for the entire variation between the three curves. It raises the question of the degree to which the characteristics or properties of a cement is a function of the chemical composition alone.

There is no doubt but that the burning temperature, the presence of small amounts of alkali and of other elements, the character of the silica, whether soluble or insoluble, the amount of calcium sulfate, the fineness of grinding, the completeness of the transformation of the tri-calcium aluminate into the Beta form, the amount of mixing water used, the presence or absence of free lime, and the conditions of storage of the briquets, will have a great influence on the components present in the cement and upon their reaction with the mixing water and consequently upon the strength and other properties of the resulting paste. The personal equation in making and testing the specimens is another variable, as is the quality of the sand used in the mortar tests. In order properly to evaluate the tensile strength of portland cement and of the factors which affect it, more information must be available. The variables just mentioned must be reduced in number and controlled so that the effect of those which remain may be determined each by itself and not as a composite of a large number of variables.

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[1] Taylor and Thompson, "Concrete-Plain and Reinforced," 2nd Ed., p. 99 (1912).

It is at once apparent when a search is made for data adapted to a study of this type that such data are not plentiful. It is possible to find many reports on strength tests and many on chemical analyses, but it is difficult to find a series of tests in which the physical tests and chemical analyses are given. In order to determine the effect of the different elements which enter into the various properties of cement and concrete material, it is becoming increasingly important that complete information be available, and it is to be hoped that some of the investigations now under way will undertake to supply this need. But, caution should be used in the interpretation of such data. It is doubtful whether the routine chemical analysis such as is made for control purposes in the mill is sufficiently accurate for the purpose outlined here; certainly where the iron and alumina are determined together and the alkali determined by difference or not at all, the analysis is of little value for scientific purposes.

This paper is not intended to present a method which may be used at the present time, under all conditions, to estimate the strength of portland cement from its chemical composition but rather to call attention to the possibility of a fairly close relationship between the two. It is another point of view from which the strength may be considered, similar in its essentials to that used by Campbell[1] and by Sauveur[2] for the estimation of the strength of steel in which the percentages of carbon, phosphorus and manganese are known. While some of the results presented here show considerable lack of agreement, and while there are many uncertainties, the close correspondence of the shape of the curve computed from the experiments made at the Bureau of Standards with that of a large series of tests, represented by the Taylor curve, is suggestive of predictions which may be made il the determining conditions are more fully known and understood.

Acknowledgment.—The author wishes to acknowledge the assistance of Assistant Professor F. E. Richart, in reviewing the manuscript and suggesting improvements therein.

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[1] H. H. Campbell, "Manufacture and Properties of Iron and Steel," 4th Ed., p. 23 (1906).

[2] A. Sauveur, "Metallography and Heat Treatment of Iron and Steel," 3d Ed., p. 66 (1926).

DISCUSSION

Mr. Bates. MR. P. H. BATES.[1]—I have not read as closely as I should this paper by Mr. Draffin, but at the same time there are a number of things that are of interest and worthy of some comment.

You will recall these comparative values are based largely on tensile strengths. I will say furthermore that the values of the Bureau of Standards are based upon rather a few number of tests—necessarily so because when we begin to make tri-calcium silicate we are in the position, so far as costs are concerned, of producing synthetic gold. By the time one has a pound of pure tri-calcium silicate, one has a material that is worth as much as a pound of gold.

However, there are possibilities in such comparisons and such studies of strength data in general. I have been applying it for a long period of years to concretes made from cements about which I knew a little more than we ordinarily do, and in which we were particular to examine microscopically and determine according to our best known methods just how these constituents were actually present and their amounts. You can derive some very interesting comparisons in that way and predictions as to what you will get in the form of concretes.

There are, of course, a number of disturbing influences as Mr. Draffin mentioned—in particular our lack of knowledge of just how the iron and magnesia are present and how they affect the constituents that are normally present. For instance, if one introduces a little iron into a mix one will immediately find that one still has apparently the same amount of pure compound produced as though no iron were present. In other words, the iron seems to enter into solid solution in silicates and modifies them to an uncertain extent. The same is true of magnesia and of the alkali. But on the other hand, such studies as this, coupled with more complete studies and a more complete knowledge of how these minor constituents affect the major constituents, will ultimately result in our ability to predict the strength value of concrete, assuming our good friend water content is constant.

It is rather annoying that others have not gone into this particular feature of cement and concrete research and studied their own product along this line. Mr. Draffin has done a nice piece of work here in indicating how such a study may be made with the briquets. There is a bigger possibility in applying that knowledge to concrete.

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[1] Chief of Division of Cement, Concrete, Lime and Ceramics, U. S. Bureau of Standards, Washington, D. C.

A MEMBER.—I should like to ask Mr. Bates if in the preparation of these individual compounds—tri-calcium aluminate, tri-calcium silicate and di-calcium silicate—it has been possible to duplicate the strength in their tests. **A Member.**

MR. BATES.—Yes. It is rather easy. The reason we commonly think we cannot do that is because we are then comparing Portland cements where there are so many other variables that we cannot do it. When we say we can, I want to except the tri-calcium silicate because if one makes several pounds of that material one is thoroughly satisfied and will not go ahead and duplicate. There it is not a question of one burn; but a question of many burns to obtain a small quantity of the product. **Mr. Bates.**

LONG-TIME TESTS OF HIGH-MAGNESIA PORTLAND CEMENTS[1]

By P. H. Bates[2]

Synopsis

This paper presents data obtained in testing a number of cements prepared in the U. S. Bureau of Standards' experimental cement plant, containing from normal to very high percentages of magnesia. The data obtained in testing at early ages and a detailed discussion have already been presented in *Technologic Paper No. 102* of the Bureau. This paper contains in addition data obtained in testing concrete made from the cements at the end of ten years. With care in manufacture, a satisfactory cement could be produced containing 6.5 per cent of magnesia. Such cements would have lower early strengths than those of lower magnesia content, but show a sufficiently marked gain in strength with age to justify classing them with normal cements at later ages. Ten-year strengths of concretes made from cements containing as much as 14 per cent of magnesia showed no marked disintegration.

Introduction

About twelve years ago the U. S. Bureau of Standards prepared, in its experimental cement plant, 18 portland cements, the magnesia contents of which varied from 1.77 to 25.53 per cent. From these cements the usual 1:3 standard sand tension briquets and 2-in. cube compression test specimens were made. These, after storage in water, were tested at ages ranging from 7 days to 3 years. There were also made a number of 6-in cubes of 1:1½:4½ gravel concrete which were tested at 4, 13 and 26 weeks and at 1½ and 10 years. These were stored either in water, in a solution of chlorides and sulfates, or in air. These data, excepting those obtained at 10 years, together with a discussion of the whole question of the effect of magnesia in cement, were published in 1918 in Bureau of Standards' *Technologic Paper No. 102.*

At that time this question of the effect of magnesia in cements on the qualities of the mortars and concretes made therefrom was a matter of considerable discussion. The standards of this Society for cement were in the course of being revised and it was very desirable to have at hand as much data pertinent to this subject as possible.

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[1] Published by permission of the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Chief of Division of Cement, Concrete, Lime and Ceramics, U. S. Bureau of Standards, Washington, D. C.

With the adoption of the revised standard permitting the use of 5 per cent magnesia (1 per cent more than in the previous standard), the interest in the matter seemed to have abated. Consequently, the Bureau did not publish the data it obtained in testing the concretes at the age of 10 years. However, recently this question was brought to the attention of this Society by a committee of another association interested in cement. It was intimated that there were no long-time data available on this subject and that an investigation should be inaugurated. It has been felt, therefore, that the presentation of the 10-year test data together with a brief description of the preparation and testing of the cement, abstracted from the above Technologic Paper, would be of value to the members of this Society. Those especially interested in the problem should also have recourse to the original publication.

PREPARATION OF THE CEMENTS

The cements were burned in the experimental rotary kiln (2 ft. in diameter by 20 ft. long) of the Bureau of Standards from raw materials the character and fineness of which were known and at temperatures which were frequently determined during the burning and maintained constant within 25° C. Two series of burnings were made, the first consisting of the production of nine cements with the magnesia content ranging from 1.77 to 18.98 per cent, and the second consisting also of nine cements with the magnesia content ranging from 2.01 to 25.53 per cent. Thus, 18 different cements burned at as many different times were investigated. All of these were of normal composition except for the magnesia content. The resulting clinker was carefully examined petrographically and the various constituents noted and their amounts approximately determined.

The raw materials were clay, limestone and dolomite in the first series, and in the second series, kaolin was used in addition to the clay in order to increase the alumina content. After cement No. 16 in which limestone alone was used as a source of the lime, increasing amounts of dolomite were used in replacing the limestone until cement No. 33 was made. In this latter, a raw mixture was used containing dolomite and no limestone. The succeeding cements burned were from raw mixtures in which increasing amounts of limestone replaced dolomite until, finally, again a raw mixture containing all limestone was used. Each cement was produced from a separate lot of raw material and was burned on a different day. Table I gives an analysis of the cements when ground to the fineness indicated in Table II, and also the average temperatures at which they were burned.

In the burning of the raw mixes, two conditions were noted that are of particular interest. Both of these would affect commercial plant production to the extent of practically limiting, from the production viewpoint alone, the maximum of magnesia permissible in a mix unless extra cost of manufacture would be permissible. The high-magnesia raw mixes gave clinker that when burned at the proper temperature as judged by the appearance alone, dusted in a very short

TABLE I.—ANALYSES OF THE VARIOUS CEMENTS AND AVERAGE TEMPERATURE OF BURNING.

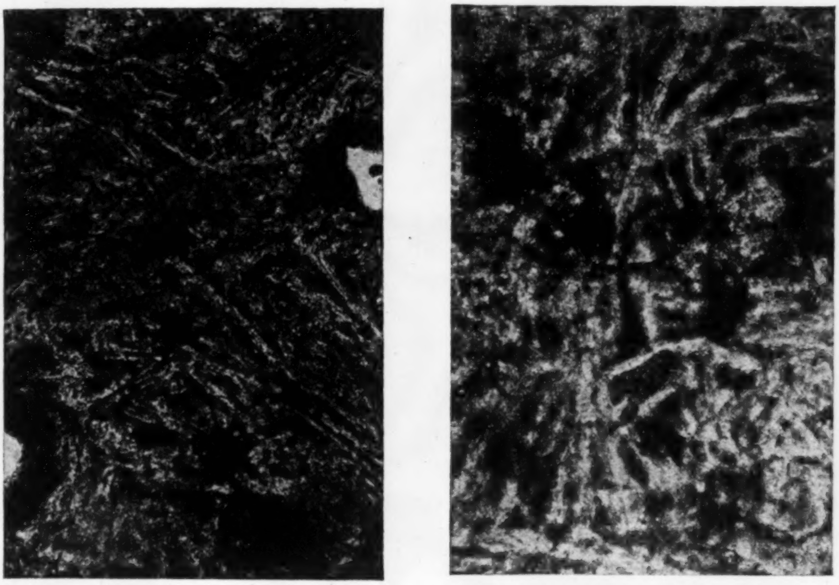
Cement	SiO_2, per cent	Al_2O_3, per cent	Fe_2O_3, per cent	CaO, per cent	MgO, per cent	SO_3, per cent	Na_2O, per cent	K_2O, per cent	Ignition Loss, per cent	Total	Average Burning Temperature, deg. Cent.
No. 16...	21.49	6.77	2.88	64.54	1.77	1.62	0.07	0.34	0.52	100.00	1520
No. 25...	21.81	7.08	2.57	62.68	3.27	1.43	0.10	0.57	0.71	100.22	1430
No. 26...	22.08	7.60	2.69	61.37	4.20	1.54	0.10	0.32	0.38	100.28	1485
No. 27...	22.08	7.51	2.94	60.16	5.12	1.65	0.12	0.26	0.36	100.20	1510
No. 28...	22.09	7.11	2.77	59.60	6.49	1.10	0.14	0.30	0.61	100.21	1505
No. 29...	22.61	7.93	2.82	56.88	7.41	1.60	0.09	0.34	0.64	100.32	1530
No. 30...	22.71	7.07	2.96	55.54	9.50	1.63	0.15	0.34	0.46	100.36	1495
No. 31...	23.12	7.67	3.31	48.00	14.07	1.58	0.18	0.41	1.35	99.69	1520
No. 32...	22.84	7.78	3.41	44.09	18.98	1.61	0.28	0.52	0.60	100.11	1500
No. 33...	22.35	8.95	3.39	36.32	25.53	1.43	0.38	0.56	1.01	99.92	1415
No. 34...	22.33	8.32	3.24	43.92	19.45	1.73	0.00	0.16	0.71	99.86	1410
No. 35...	22.11	8.79	3.10	49.30	13.96	1.83	0.09	0.17	0.68	100.03	1420
No. 36...	21.76	8.50	3.16	53.77	10.33	1.61	0.13	0.27	0.47	100.00	1435
No. 37...	21.53	8.32	3.19	56.30	8.03	1.62	0.06	0.23	0.59	99.87	1425
No. 38...	21.39	7.85	3.43	57.26	7.80	1.67	0.09	0.38	0.55	100.42	1435
No. 39...	21.38	8.36	3.15	58.02	6.62	1.62	0.03	0.15	0.61	99.94	1450
No. 40...	21.22	8.46	3.27	61.40	3.59	1.56	0.00	0.16	0.67	100.33	1445
No. 41...	21.60	8.15	3.35	62.67	2.01	1.63	0.06	0.08	0.42	99.97	1445

TABLE II.—TIME OF SET, CONSISTENCY, AND FINENESS OF THE VARIOUS CEMENTS.

Cement	MgO, per cent	Normal Consistency	Time of Set								Passing 200 Mesh, per cent
			Vicat				Gilmore				
			Initial		Final		Initial		Final		
			hours	minutes	hours	minutes	hours	minutes	hours	minutes	
No. 16........	1.77	20.0	0	55	6	40	3	00	8	05	79.6
No. 25........	3.27	21.0	2	15	6	10	3	05	8	30	75.8
No. 26........	4.20	20.5	2	15	7	30	3	45	9	15	79.8
No. 27........	5.12	19.0	2	40	6	15	4	00	8	30	78.6
No. 28........	6.49	21.0	1	35	5	10	2	35	6	30	77.6
No. 29........	7.41	20.5	1	20	6	30	4	25	9	00	76.0
No. 30........	9.50	19.0	1	00	9	30	3	20	13	00	73.2
No. 31........	14.07	18.0	1[a]	05	1[a]	25	0[a]	06	2[a]	05	79.0
No. 32........	18.98	31.0	1[a]	00	30[a]	00	5[a]	00	48[a]	00	90.4
No. 33........	25.53	18.5	0[b]	30	25[b]	00	4[b]	00	48[b]	00	81.4
No. 34........	19.45	24.0	0[a]	05	0[a]	15	0[a]	10	1[a]	30	76.0
No. 35........	13.96	24.5	0[a]	05	0[a]	12	0[a]	08	1[a]	25	79.2
No. 36........	10.33	18.5	1	30	3	30	2	00	4	00	78.8
No. 37........	8.03	19.0	2	10	5	00	3	15	5	50	78.4
No. 38........	7.80	19.0	2	20	4	10	3	05	5	00	79.0
No. 39........	6.62	19.5	1	55	4	15	3	35	5	25	80.1
No. 40........	3.59	19.5	1	35	4	00	2	10	4	45	78.0
No. 41........	2.01	19.5	1	35	5	00	2	05	5	40	78.6

[a] All test pieces became quite warm, especially the Vicat. [b] Flash set.

time (5 to 10 minutes, depending upon the rate of cooling). If the temperature was increased until a decidedly over-burned or very hard clinker was obtained, dusting again resulted. At a temperature somewhat lower than the latter a clinker was obtained that showed minimum immediate dusting, though it slowly changed to thepowdered condition. Therefore, the temperatures at which the higher magnesia cements were burned as shown in Table I, are decidedly beyond those required to combine the basic and acidic components, but gave the maximum amounts of satisfactory clinker. This slow dusting was not noted in any of the burns containing less magnesia than that shown by No. 30 (9.5 per cent).



(a) Cement No. 37; MgO = 8.03 per cent

(b) Cement No. 40; MgO = 3.59 per cent.

FIG. 1.—Sections of Clinker Showing Marked Acicular Development of $3CaO \cdot SiO_2$ (× 275).

In usual cement-mill practice, where complete analyses of the raw mixes are not made frequently, and where the magnesia content may reach a fairly high amount, the latter condition is first noted in the kiln by a decided tendency of the clinker to "ball" or form "logs," due to the lowering of the softening point of the mixture. In this investigation this was very marked when the percentage of magnesia was increased beyond 9.5 per cent. When the amount was less, the clinker was both approximately normal in appearance and normal in its behavior in the kiln, giving the usual black, glistening, coke-like material, which rolled about in the kiln without any tendency to form balls or "logs" or to "ring-up." When this amount was exceeded, the clinker was of a light brown appearance, which gave a considerable amount of an ochre-like dust and a yellow cement, the latter giving a yellow, muddy, plastic concrete. The formation of rings was also excessive.

CHARACTER OF THE CLINKER

As noted above, macroscopically the clinkers of the high-magnesia cements were distinctly different from normal clinker. Microscopically they were also different. Normal clinker is usually characterized by an absence of distinct outline of grains. The several constituents seem to blend into one another. Less basic mixtures or highly burned ones will produce clinkers which show occasionally distinct separation of the grains. The cements in this investigation showed, however, a marked development of large grains and in all the clinker, including that of higher magnesia content, there was a marked development of acicular crystals of the tricalcium silicate (see Fig. 1).

No constituents other than those noted in normal cements were observed until the magnesia content reached 9.5 per cent. Then the mineral monticellite, $MgO \cdot CaO \cdot SiO_2$, was noted. This increased until in the cement made using dolomite alone it was present to the extent of somewhat more than 20 per cent. Its presence was accompanied by a marked decrease in the amount of the dicalcium silicate present. A very small amount of spinel, $MgO \cdot Al_2O_3$, was also noted in these very high magnesia cements.

GENERAL PROPERTIES OF THE CEMENTS

The time of set, which is shown in Table II, was of particular interest. Reference was made above to the fact that the high-magnesia content would result in increased cost of production due to the lower output per kiln, caused by the tendency of clinker of such composition to form "logs," "balls" and "rings" in the kilns. The time of set also indicates that the high-magnesia content could not be reached without entailing on the producer some changes in his present procedure which would result in increasing the time of set. From Table II it can be seen that a clinker containing about 10 per cent of magnesia produces a cement which develops a very short time of set. In fact, cements Nos. 31 to 35, inclusive, had a very quick or flash set. In the case of No. 32 an excess of water was used in order to delay the set long enough to obtain a test piece.

The explanation of this tendency is given in the original text in the chapter dealing with the petrographic analyses of the clinker. Briefly the microscopic examination showed that the amounts of quick-setting constituents (tricalcium aluminate and tricalcium silicate) in all the clinker was surprisingly constant, ranging from 38 to 61 per cent, but the amount of the slow-setting constituent (dicalcium silicate) ranged from 13 to 47 per cent. Furthermore, it was noted that as the magnesia increased the amount of the slow-hardening constituent was materially reduced, the inert non-hardening monticellite having been formed. The very minor formation of the inert spinel in the very high magnesia cement reduced the amount of quick-hardening aluminate but to a very slight degree. Apparently, therefore, those compounds which in normal portland cement reduce the activity of the quick-hardening constituents had been reduced

TABLE III.—TENSILE AND COMPRESSIVE STRENGTHS OF 1:3 STANDARD MORTARS.
All Values are Pounds per Square Inch.

Cement	MgO, per cent	Method	Tested at						
			24 Hours	7 Days	4 Weeks	13 Weeks	26 Weeks	1 Year	3 Years
No. 16	1.77	Tension.............	72	291	322	410	365	414	376
		Compression.........	398	2030	3516	4522	4199	4666	4329
No. 25	3.27	Tension.............	156	193	281	363	362	347	203
		Compression.........	712	1284	1999	2156	3204	3374	2800
No. 26	4.20	Tension.............	128	291	336	403	445	421	342
		Compression.........	555	1461	2725	3353	3761	4088	4200
No. 27	5.12	Tension.............	108	275	445	443	480	459	408
		Compression.........	515	1326	3042	3379	4754	3896	4363
No. 28	6.49	Tension.............	99	212	339	381	321	336	242
		Compression.........	640	1380	2504	3548	3715	3603	3882
No. 29	7.41	Tension.............	91	149	341	427	430	376	274
		Compression.........	395	879	1708	2618	3368	3688	3963
No. 30	9.50	Tension.............	58	110	217	378	367	355	141
		Compression.........	227	454	1324	1809	2977	2707	2633
No. 31	14.07	Tension.............	29	52	79	160	186	224	224
		Compression[a]........	...	....	....	....	....	....	....
No. 32	18.98	Tension.............	24	26	27	68	102	[b]	[b]
		Compression.........	117	133	140	288	612	442	[b]
No. 33	25.53	Tension.............	Soft	Soft	38	87	73	116	[b]
		Compression.........	27	43	110	148	434	479	[b]
No. 34	19.45	Tension.............	33	42	41	93	130	145	[b]
		Compression.........	147	137	545	896	1666	2374	[b]
No. 35	13.96	Tension.............	69	65	55	134	150	82	[b]
		Compression.........	494	396	621	873	1628	1018	[b]
No. 36	10.33	Tension.............	127	115	234	328	440	398	107[c]
		Compression.........	318	455	1394	2216	2563	2802	1154[c]
No. 37	8.03	Tension.............	90	160	305	448	400	375	168[c]
		Compression.........	386	844	2332	3550	3425	3193	3070[c]
No. 38	7.80	Tension.............	112	168	252	366	418	319	282
		Compression.........	489	911	1838	2018	2567	2986	2046
No. 39	6.62	Tension.............	155	212	345	472	461	500	355
		Compression.........	653	928	2227	1939	2792	4508	2207
No. 40	3.59	Tension.............	136	274	368	428	431	407	....
		Compression.........	568	1585	2631	2697	3896	4100	2738
No. 41	2.01	Tension.............	133	289	406	449	470	422	420
		Compression.........	673	1857	2696	3320	2758	4383	3315

[a] No test piece made.
[b] Disintegrated.
[c] Specimens were unusually full of crystals, but no enlargement or checking noticed.

through the formation of inert compounds to such a degree that quick setting resulted.

All of the cements were tested for soundness according to the present method of steaming pats, and also by the other methods then in use, namely, boiling pats at the end of 24 hours and storing them in air and in water for 28 days. All test pieces stored in the water for 28 days were sound. The test piece of No. 33 was slightly warped after 28 days in the air. All others were sound in this test. The pats from Nos. 32 and 33, not having set in 24 hours, were still soft after removal from the boiling and steaming tests. All the other pats passed these tests satisfactorily excepting those from Nos. 25 and 26. These were disintegrated in both tests. Incidentally they were low-magnesia cements in which the petrographic analyses showed approximately 3.5 and 2.5 per cent free lime, respectively. The effect of the unsoundness of these two cements is of particular interest when studied in connection with the tests of the concrete made from them. Since the concrete after 10 years aging was thoroughly sound and of excellent strength, apparently from these tests one might ignore our present soundness tests entirely.

STRENGTH

The data obtained in testing the 1:3 standard sand briquets in tension and 2-in. cubes in compression are given in Table III. The last age at which any number of these specimens was tested was three years. All the specimens were stored in water for this entire age after the first 24 hours in the molds in the damp closet. Neat briquets and cubes were also tested at these same ages. For data concerning them reference must be had to the Technologic Paper. Judging from our present A.S.T.M. standards a magnesia content exceeding 6.5 per cent cannot be exceeded if a product is to be obtained which will meet these standards for strength and set. However, it can readily be seen that at 13 weeks the slower-hardening cements containing as much as 10 per cent magnesia had developed most satisfactory strengths, comparable with those developed at this age by the lower-magnesia cements. Too little information is available as to what should be the strength of a normal cement as a mortar in compression to permit of much of a discussion of the compression values obtained in this investigation. It can be seen that at 7 or 28 days the cements containing more than 6.5 magnesia are quite low in compressive strength, but at 3 or 6 months those containing approximately 10 per cent have sufficient strength to place them in the class of acceptable cements.

The data obtained from the 6-in. cubes of 1 : 1½ : 4½ gravel concrete are given in Table IV. The storage of these specimens was rather different from that generally used in aging concrete. This procedure was followed largely because of the arguments found in the literature regarding the deportment of cements of the compositions under study. Thus it has always been stated that cements containing much magnesia should not be used when products made therefrom would be subject to sea-water action. Specifications in use at that time in this country had limited the magnesia content of cements for such purposes to a maximum of 3 per cent. It has also been stated that the high-magnesia cements would disintegrate more quickly when stored in water than in air. Hence, two types of storage were used. Half the specimens made from each cement, after removal from the molds at 24 hours, were placed in water and the other half in a 2-per-cent solution composed of equal parts of crystallized sodium chloride, sodium sulfate,

TABLE IV.—COMPRESSIVE STRENGTH OF 1:1½:4½ GRAVEL-CONCRETE CUBES.
All Values are Pounds per Square Inch.

Cement	MgO, per cent	Stored in	Tested at				
			4 Weeks	13 Weeks	26 Weeks	1.5 Years	10 Years
No. 16	1.77	Water	3491	4067	4081	6133	4970
		Solution	3388	3829	3435	6245	4850
No. 25	3.27	Water	2910	3625	3765	5084	4020
		Solution	2932	3756	3951	4996	4890
No. 26	4.20	Water	3275	3776	4379	6238	4530
		Solution	3351	4261	4564	6558	5170
No. 27	5.12	Water	3410	4228	4605	6528	4685
		Solution	3427	4194	4318	6276	5080
No. 28	6.49	Water	2489	3493	4185	5843	5205
		Solution	2466	3173	3771	5875	4780
No. 29	7.41	Water	2059	3171	3431	5742	4385
		Solution	1675	2813	3119	4650	4090
No. 30	9.50	Water	1616	2254	2589	4610	3595
		Solution	1305	2343	2563	4068	3710
No. 31	14.07	Water	483	1040	1248	2608	2200
		Solution	533	1342	1488	2989	2550
No. 34	19.45	Damp closet	133	476	787	1589	....
		Air	63	105	77	100	....
No. 35	13.96	Water	408	768	1044	2062	2095
		Solution	308[a]	833	1127	2112	2205
		Air	320	378	512	532	885
No. 36	10.33	Water	1174	1868	2263	3903	3690
		Solution	1003	1731	1917	3138	2495
		Air	1271	1957	2002	2552	3010
No. 37	8.03	Water	1660	2580	2714	4454	3675
		Solution	2008	2611	2744	3832	3835
		Air	964	1262	1307	1657	2560
No. 38	7.80	Water	1836	2587	3232	4878	....
		Solution	1794	2624	3192	4697	....
		Air	1094	1404	1473	1604	....
No. 39	6.62	Water	2261	3406	3142	4735	4120
		Solution	1972	3330	3381	4186	3850
		Air	1260	1362	1454	1562	2515
No. 40	3.59	Water	2286	2859	3037	4363	3550
		Solution	2364	3032	3440	4522	3560
		Air	1478	1612	1524	1838	2430
No. 41	2.01	Water	2416	2887	3206	4561	3690
		Solution	2045	2729	3180	4348	3465
		Air	1651	1800	1899	1882	2325

[a] Wet when tested.

magnesium chloride and magnesium sulfate. This latter was used in lieu of sea water, particularly because previous work[1] had shown that such salts were most active in disintegrating concrete. Furthermore, the procedure of alternate immersing and drying was followed, since this tends towards more rapid destruction than continuous immersion. For the first 13 weeks the periods of immersing and drying were 1 week each. Between 13 and 26 weeks the periods of immersion had been increased to 3 or 4 weeks with intermediate periods of drying of 1 week. Between 26 weeks and 1 year the immersion periods were 4 weeks with intermediate drying periods of 2 weeks. At the end of 1 year all specimens were stored continuously in the air of the laboratory. A set of specimens of cements No. 35 to 41, inclusive, were also stored throughout their life in the laboratory air. The setting characteristics of cements Nos. 31 to 35, inclusive, hardly justified the making of concrete specimens and hence specimens were made of but three of them. They required long aging in the molds before sufficient strength was developed to permit of handling and even then the specimens from No. 34 were not strong enough to permit of storage in the water or the solution.

Again it is seen that regardless of the method of curing, cements containing 6.50 per cent magnesia are quite satisfactory at four weeks. At later periods the strength of the concrete from the cements containing approximately 10 per cent magnesia is also good. The retrogression between the 1½-year period and the 10-year period is common to all of the cements, regardless of their composition, if stored in water or solution. Note, however, that there is no retrogression between these ages when the specimens are stored continuously in air but, on the other hand, there is a constant gain in strength. It would seem, therefore, logical to conclude that the decrease of strength was due to the change of storage conditions, as indicated above, at the end of a year. It is not as well known as it should be that the maximum strength of concrete is developed at some age in the presence of a certain optimum amount of water, which may be held either mechanically or in the several chemical ways. A specimen tested immediately on removal from a damp closet or water is not as strong as one subjected to a certain amount of drying. At the same time a long continuous drying may produce a lower strength than no drying. It apparently happened that removal from the water or solution at the end of one year and testing at the end of 1½ years gave the amount of drying that was required for a high strength. The storage in the air until the testing at ten years resulted in too large a degree of dessication and a consequent lowering of strength. The similarity of the bonding produced by an organic glue and by cement must be borne in mind. In the former, the maximum bond is produced by an optimum water content. Too much water, possibly due to too great a humidity in the surrounding atmosphere or too little water due to too low a humidity, will result in a lowering of the bond. The same conditions hold in the case of the bond produced by portland cements.

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[1] U. S. Bureau of Standards *Technologic Paper No. 12*, "The Action of the Salts in Alkali Water and Sea Water on Cements."

CONCLUSIONS

Certain of the conclusions appearing in the Technologic Paper which would interest those concerned with the physical qualities of cement rather than with the chemical characteristics are presented below. While these conclusions were based upon the deportment of the cements at the end of 1½ years, the results obtained at ten years do not justify any changes. It should be borne in mind that the question of the magnesia content of cement is a large and intricate one and was not completely covered in this investigation. Thus it can be readily appreciated that the difference in the molecular weights of lime and magnesia would have required changes in the amounts of the acidic components of the raw materials as the ratio of lime and magnesia was changed. This was not done in this investigation. The procedure followed was that more commonly used in commercial practice, namely, holding the ratio of total basic to acidic components constant.

1. "Portland cement with a magnesia content of about 9.50 per cent may be burned in a rotary kiln without producing a clinker materially different from one containing less than 4 per cent. The clinkering temperature will be reduced somewhat, however. With greater amounts of magnesia present the resulting clinker is very vitreous and dusts more or less slowly, the rapidity and amount of dusting increasing with the magnesia content. High-magnesia clinker is of a light-brown color, in strong contrast to the usual dark, glistening, normal clinker. The resulting ground cement is of a light-brown color, which makes a concrete decidedly different in color from concrete made from normal cement."

2. "The strengths developed, either by the neat cement or 1:3 sand mortar or 1:1½:4½ gravel concrete, show that cements containing as much as 7.5 per cent of magnesia are satisfactory. It would be impossible to predict from the strength tests at the end of 1½ years which were the cements containing low magnesia or magnesia up to 7.5 per cent. With higher amounts the strengths developed decreased

with increased magnesia, but even with the high-magnesia cements there is a notable increase of strength with age."

3. "The strength of the concretes subjected to the action of the solution of salts was not materially different from that of those subjected to the action of water. While some of the specimens did show a slight disintegration, it was not sufficient to affect their strength at the last period at which they were tested, and, furthermore, it appeared in the case of concrete made from cements of moderate magnesia content and was not noted in those of high-magnesia content."

4. "The high-magnesia cements contain a large amount of 'insoluble residue.'[1] This residue is the monticellite and spinel, both of which are very largely insoluble in dilute acids."

"Attention should again be called to the fact that the work presented in this paper was not undertaken to show the advisability of allowing a greater magnesia content in cement, but only to determine how greater amounts of magnesia affect the constitution and properties of cement of normal composition."

In the above quoted conclusions from the Technologic Paper, it should be noted that it is stated that a magnesia content of 7.5 per cent would not be too high to permit of a cement having satisfactory qualities. However, the recent modification of the Society's standards for portland cement would indicate that 6.5 per cent would be the maximum, if these strength requirements are to be met as indicated elsewhere in the text.

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[1] A.S.T.M. Standard Specifications and Tests for Portland Cement (C 9 - 26), 1927 Book of A.S.T.M. Standards, Part II.

SYMPOSIUM ON FIELD CONTROL OF THE QUALITY OF CONCRETE

INTRODUCTION

By Cloyd M. Chapman[1]

It has become increasingly difficult during the past few years for engineers, architects, contractors, construction superintendents and others interested in the making of concrete, to keep up with the times,—to keep informed of the latest developments in the art and the science of making concrete. So many new theories have been advanced, so many improvements made in the various steps of the process of producing good concrete, and so many novelties of various sorts have been introduced,—devices, equipment, admixtures, methods of test,—that unless one is intimately associated with the forces which are bringing about these changes, or keeps very closely in touch with the literature of the subject, it is difficult to form opinions of or pass judgment on many of these matters.

Changes and improvements have appeared faster than the industry could assimilate them. Theories of design of mixes have been advanced, one after another. Before information concerning one new theory could be spread abroad and discussed, another came into the spotlight. Admixtures by the score have appeared. Proportioning machines, weighing and measuring devices, mixing and transporting equipment, all have sprung up either as new tools for the concrete maker or as old tools in a new form. It was but eleven years ago, in 1916, that the proposal was made that the arbitrary-mix method of specifying concrete be abandoned and a mix specified which upon trial test had developed the strength required by the design. It was but nine years ago that the fineness-modulus method of designing concrete mixtures was proposed and the relation between the water-cement ratio and strength of concrete was shown. The first slump test was made in 1912. Sand was measured under water to secure constant volume in a sand tester brought out in 1916. Light-weight concrete of high strength and impermeability was a novelty developed during the World War and is therefore only about ten years old. High-early-strength cements have been commercially developed in this country only within a very few years, although they have been in use for a number of years abroad. Each and every one of these developments, whether of theory or practice or tools or materials, bears a definite relation to the quality of the concrete produced. What are these relations? And how may they be utilized to insure better concrete? Following a certain procedure with certain equipment may produce a large volume of concrete in a given time and do it at a low cost, but what about the *quality* of the concrete? A concrete mix may have been carefully and well designed in the laboratory; but what will happen to the *quality* of the concrete if, in the field, the design is not closely followed? These and similar questions are continually coming up in the everyday work of the concrete maker. And not all are agreed on the answers to many of these questions. Long and lively and without apparent change in conviction by either party, have been some of the arguments thereon. But discussion breeds ideas, and we need ideas.

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[1] Consulting Materials Engineer, Dwight P. Robinson and Co., Inc., New York City.

This collection of papers has been brought together with a view to presenting as complete and continuous a story as the limitations of time and space will permit of the factors which affect the quality of concrete and which are subject to regulation or control in the field. The story starts with the design and proportioning of the mix, since this phase of concrete-making often must be conducted in the field or at least be subject to review and revision under changing field conditions. Then, in order, follow mixing, conveying and placing, provision for expansion and contraction, and protecting from low temperatures. To know the quality of the day-to-day output one must make tests, and therefore two papers are included on this very vital subject. It is not pretended that the story is complete, nor that it is the last word on any phase of the subject. Some may not agree with some of the statements made. The discussion will indicate the nature and extent of the disagreement.

The authors of these papers were deliberately and carefully chosen, and the subject and scope of each paper were defined in advance of its preparation. There are, however, particular phases of the broad subject which are not as fully treated as they deserve. For example, there is the matter of the improved quality obtainable by vibrating forms during or immediately after placement of the concrete. There were many valuable data gathered on this subject during the War in the construction of concrete ships. The removal of surplus water after placing concrete in the forms is another matter worthy of consideration. It is hoped and expected that these and other matters which are lightly touched upon by the authors, will be more fully covered by the ensuing discussion, which will constitute just as important and valuable a part of the story as the more formally scheduled papers.

PROPORTIONING OF CONCRETE

By R. W. Crum[1]

Synopsis

The paper discusses the factors due to composition that affect the strength of concrete and the methods for design of mixtures based thereon. It is shown that both the water-cement ratio and the void-cement ratio are indices of the crushing strength of concrete. Concrete mixtures can be designed either by the water-cement ratio method or by the mortar-void method with reasonably concordant results. The paper points out that, for accurate results in specific cases, the design of mixtures by either theory should be based upon preliminary tests of the materials to be used, under conditions comparable to the expected field conditions. Practical examples are worked out in the paper.

The paper then discusses the details of field operations required in putting the water-cement ratio method of control to practical use. Attention is called to needed improvements in water-measuring apparatus. Measurement of aggregates by weight is described and its advantages discussed, including the advantages to the contractor and the benefits resulting from improved concrete.

PART I.—DESIGN OF MIXTURES.

In so far as it is affected by the proportions of the constituent materials, the average quality of concrete is governed in the field by the control of the water content of the batch. Variation from the average or uniformity as affected by the mix is controlled by the accuracy of measurement of the various ingredients.

Two basic facts have become commonly accepted.

1. The quality of concrete depends upon the ratio of the void space in the concrete to the cement.

2. The quality of concrete depends upon the ratio of mixing water to cement.

These factors must necessarily be related, and the following analysis of the effect of composition on quality is based thereon.

The quality of concrete made from sound, durable materials depends primarily upon the actual amount of solid materials present, and the extent to which the particles of solid matter are glued together. If it be assumed that the void space in the concrete is a function of the amount of material, and that the amount of cement is a function of the cementing action, the ratio of voids to cement may be expected to be indicative of quality. In fact, numerous investigators have shown that strength varies with the ratio of voids to cement. It follows, therefore, that for a given quantity of cement per unit of volume of concrete, maximum strength can be expected when the void space is a minimum. It has been found that the volume of void space in plastic mixtures is practically controlled by the amount of mixing water, and that, therefore, the ratio of mixing water to cement is also indicative of the quality of the concrete. This has been repeatedly demonstrated.[1]

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[1] **Engineer of Materials and Tests, Iowa State Highway Commission, Ames, Ia.**

Many series of tests have been studied. In all of these, when the data are plotted using compressive strength as one ordinate and water-cement ratio or void-cement ratio as the other, the points indicate a tendency which can be represented by an equation of the form $S = \frac{B,}{A^x}$ in which S = compressive strength in pounds per square inch, x = cubic feet of mixing water or cubic feet of voids per bag of cement, and B and A are empirical constants. This expression was first suggested by D. A. Abrams.

In studying test data from a number of different sources,[2] it is noticeable that although a curve may be found to represent each series of tests, the different sets of tests give different positions of both the water-cement and void-cement curves. Doubtless many variable factors are responsible for this condition. Different cements, aggregates of different physical characteristics, different molding methods, and different curing conditions are some that come readily to mind.

It is also noticeable that in any series of tests, there is considerable variation in individual strengths. Inaccuracies inherent in our present methods of testing will account for a part of these variations. In the case of the water-cement relation doubtless some variations are due to differences in the extent to which the void spaces are filled with water. The data[3] reported by the University of Texas in tests sponsored by Committee C-9 on Concrete and Concrete Aggregates, Series 201, are interesting in this connection. Two sets of test specimens were made under identical conditions except that one set was rodded a number of times, to drive out air and excess water. These data have been plotted in Fig. 1, and it will be noticed that the water-cement-ratio-strength curve for the rodded concrete is higher than for the standard molded concrete, although the curves are practically parallel. Since some of the water was driven out by the rodding,

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[1] For data demonstrating this fact, reference is made to the current report of Committee C-9 on Concrete and Concrete Aggregates, particularly to the paper included therein by R. A. Nelson on "A Study of the Data of 'Series 201' and Wisconsin Tests Showing the Relation of the Compressive Strength of Concrete to the Water-Cement Ratio, Space-Cement Ratio and Grading of the Aggregate," see *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I, p. 361 (1927).

[2] See Fig. 1 in paper by R. A. Nelson on "A Study of the Data of 'Series 201' and Wisconsin Tests Showing the Relation of the Compressive Strength of Concrete to the Water-Cement Ratio, Space-Cement Ratio and Grading of the Aggregate," *Ibid.*

[3] Report of Committee C-9 on Concrete and Concrete Aggregates, Appendix II, "Summary of Data Obtained in 'Series 201,' Compression Tests of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 329 (1922).

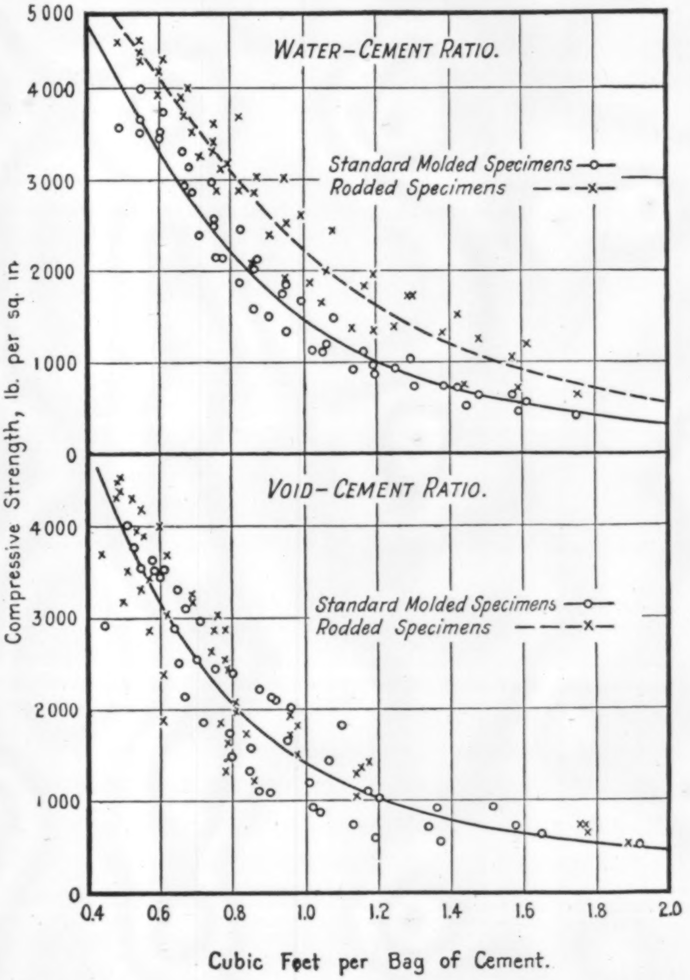

FIG. 1.—Effect of Rodding upon Water-Cement and Void-Cement Ratios and Compressive Strength. Data from Tests by University of Texas, A.S.T.M. Committee C-9, "Series 201."

the water ratios applied to the rodded concrete must be in error. If the correct values for the water ratios were known and plotted, this curve would be moved to the left and would probably come into

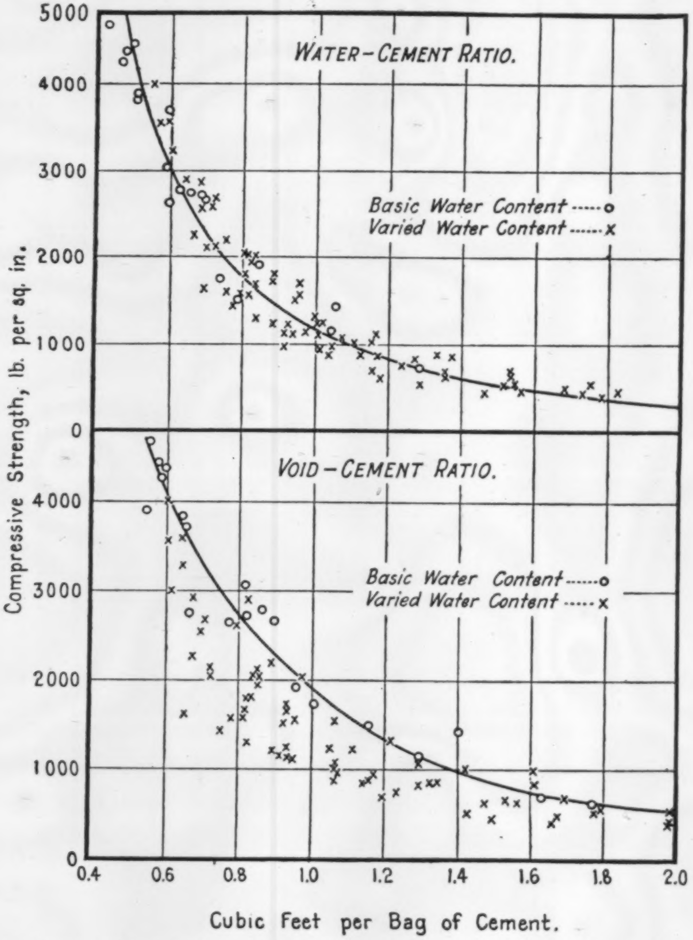

FIG. 2.—Effect of Varied Relative Water Content upon Water-Cement and Void-Cement Ratios and Compressive Strength. Data from Tests of Talbot and Richart, *Bulletin No. 137*, University of Illinois.

coincidence with the other. It may also be seen in Fig. 1 that the data for both sets of tests conform to the same void-cement curve, which is as it should be since the correct values for voids and cement could be determined.

There is some evidence to show that water has an effect upon quality beyond its effect in controlling the size of the voids. In their bulletin[1] on "The Strength of Concrete, its Relation to the Cement, Aggregates and Water," Talbot and Richart demonstrate from one series of tests (shown in Fig. 2) that the maximum strength for a given void-cement ratio will be secured when the amount of water used is

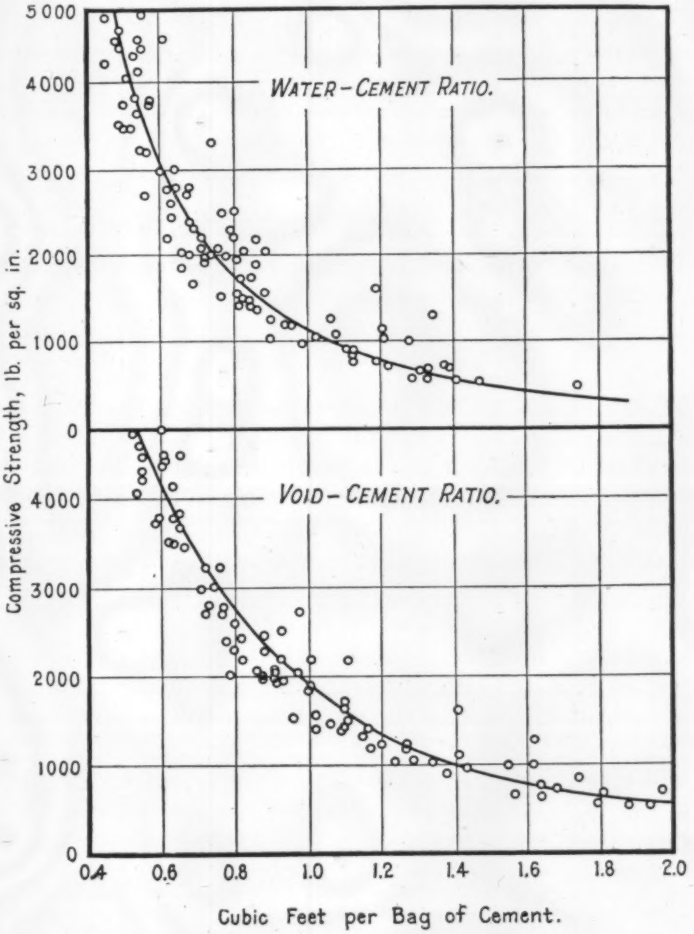

FIG. 3.—Water-Cement and Void-Cement Ratios and Compressive Strength of Mortar at Basic Water Content. Data from Tests of Talbot and Richart, *Bulletin No. 137*, University of Illinois.

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[1] *Bulletin No. 137*, Engineering Experiment Station, University of Illinois.

that which will yield the smallest volume of mortar when mixed with a certain amount of cement and fine aggregate. This particular amount of water they call "basic water content." In these tests when more water was used than needed for basic water content, the concrete was lower in strength even though the void-cement ratios were the same. It may also be noted in Fig. 2 that these data show the same characteristic water-cement ratio curves as other data, and that apparently the effect of using water in excess of the basic amount is automatically accounted for in the water ratio curve. Figure 3 shows the characteristic curves for concrete at basic water content, from data by Talbot and Richart.

Undoubtedly more information is needed as to the variables that cause individual variations from the basic relationships between mixing water, voids and cement.

THEORIES FOR DESIGN

The use of the water-cement ratio as the principal factor in designing concrete mixes was first proposed by D. A. Abrams[1] in 1918, and has since that time been frequently recognized in specifications by the requirement that the consistency be as dry as conditions of workability permit. Conscientious administration of such a specification would no doubt produce concrete of average maximum strength for the amount of cement used, but since a comparatively small change in water ratio may mean a considerable decrease in strength, it would seem much better to specify also the ratio of water to cement. According to F. R. McMillan, "Designing a concrete mix for a given strength consists in selecting the water-cement ratio corresponding to that strength and finding the most suitable combination of aggregates which will give the desired workability when mixed with cement and water in this ratio."

It should be borne in mind that probably the very best results will be secured if the stiffest consistency compatible with plasticity and workability is adopted. The water-cement ratio will undoubtedly establish the class of the concrete, but in order to obviate so far as possible the variations in tests of individual specimens, each batch as made should have exactly the same ingredients as every other batch.

The method for designing mixtures proposed by Talbot and Richart[2] takes into account not only the effect of water upon voids but also the additional effect upon strength of using water in excess of the basic water content. This method of designing concrete mixtures is based upon the principle that the strength of concrete depends upon the ratio of voids to cement in a given volume of concrete, and also that the greatest strength will result for a given ratio of voids to cement when the mortar part of the concrete has "basic water content." This assumption is based upon the data depicted in Figs. 2 and 3, which show the discrepancy in the void-cement-strength relation, when water contents other than basic are used.

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[1] *Bulletin 1*, Structural Materials Research Laboratory, Portland Cement Association and Lewis Institute.

[2] A. N. Talbot and F. E. Richart, "The Strength of Concrete, Its Relation to the Cement Aggregates and Water," *Bulletin No. 137*, Engineering Experiment Station, University of Illinois.

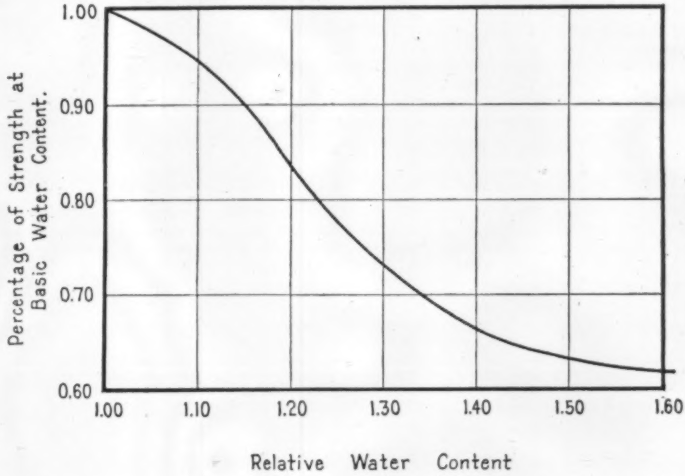

FIG. 4.—Reduction Curve for Concretes of Varied Water Content. Data from Tests of Talbot and Richart, *Bulletin No. 137*, University of Illinois.

Talbot and Richart state:

> "The discrepancy is small at relative water contents (water content in percentage of basic water content) close to basic, but it increases as water is added. It may be considered that there are two sources of the lower strengths of such concretes: (*a*) the increase in the voids which comes with an increase in the water, and (*b*) an additional effect of the presence of excess water. It would appear that the decrease in strength due to the presence of the excess water is about the same as, or somewhat more than, that due to the increase in voids."

Based upon these data a reduction curve was devised by Talbot and Richart for use in designing mixtures according to their method, shown in Fig. 4.

> "This reduction curve means that at a relative water content of say 1.3 the strength of the concrete is 0.73 of that which would correspond to the same

voids at basic water content. Conversely the strength at basic water content for a given value of the voids may be obtained by dividing the strength for the given water content by a ratio taken from the reduction curve."

In this method of design the assumption is also made that the strength of the concrete depends upon the strength of the mortar, if the mortar more than fills the voids in the coarse aggregate. It is assumed that the particles of stone merely serve as filling in a matrix of mortar and, therefore, the absolute volume of the voids in the concrete is equal to the absolute volume of the voids in the mortar.

TABLE I.—CONCRETE TEST SPECIMENS FOR DESIGN OF CONCRETE MIXTURES, IOWA HIGHWAY COMMISSION SERIES R-48.

Ratio of Fine Aggregate to Coarse Aggregate, 33:67 by weight.
Fine Aggregate: Sand graded 0 to No. 4: Fineness Modulus 3.25.
Coarse Aggregate: Gravel graded No. 4 to $1\frac{1}{2}$-in. opening; Fineness Modulus 7.48.
Each value is the average of 12 specimens.

Ratio Aggregate to Cement, by Weight	Absolute Volume per Unit of Volume					Cubic Feet of Water per Bag of Cement	Crushing Strength, lb. per sq. in.		Cubic Feet of Voids per Bag of Cement	Relative Water Content	Slump, in.
	Water	Cement	Sand	Gravel	Voids		7 Day	28 Day			
6.25	0.154	0.102	0.248	0.515	0.135	0.75	1973	3575	0.642	1.42	1.46
8.93	0.153	0.075	0.257	0.532	0.134	1.00	922	1891	0.867	1.62	1.25
10.77	0.161	0.062	0.259	0.537	0.143	1.25	522	1274	1.125	1.65	1.40
14.03	0.150	0.048	0.263	0.546	0.142	1.50	393	976	1.535	1.70	1.50

DESIGN PROCEDURE

For designing according to the water-cement-ratio method proposed by Abrams, all that is needed is the relation between strength and various water-cement ratios. For the Talbot method the mortar-void characteristics of the particular sand to be used are needed, as well as the relation between strength and various ratios of voids to cement. Abrams has suggested an equation, $S = \frac{14000}{7^x}$, for the strength-water-cement relation and Talbot has suggested a curve for the strength-void-cement ratio which may be generally applicable. However, since other sets of tests have given other values for the constants in Abrams' formula, and other positions of the void-cement-strength curve, it would seem to be better practice, in applying either of these methods, to base the results upon curves developed especially for the conditions at hand. According to the most recent commonly accepted basis for design of concrete mixtures, the problem, therefore, is essentially one of field control.

Design of mixtures as respects quality only, by both of the above methods, is worked out in the following examples, both being taken from data covering the same materials. The tests made were such as would have to be made in the application of these designing methods. The economic aspects of variations in grading of aggregates will not be considered in this discussion.

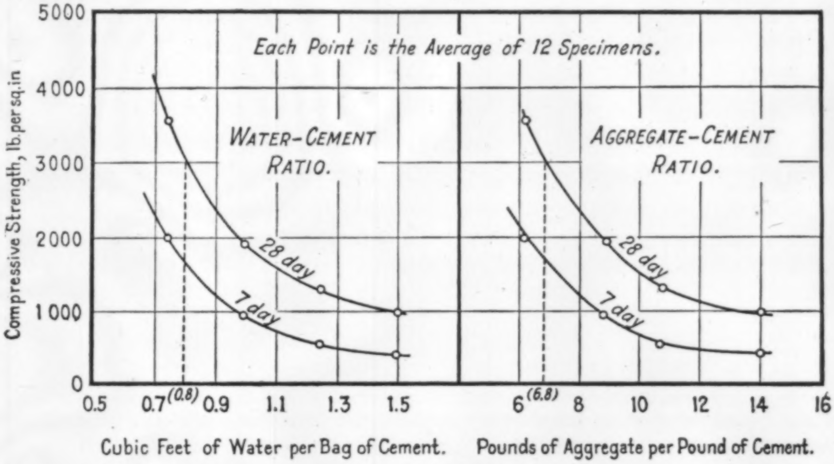

FIG. 5.—Water-Cement Ratio and Aggregate-Cement Ratio and Compressive Strength of Concrete. From Tests for Design of Mixtures, Iowa Highway Commission, Series R-48.

For use in these examples a series of specimens was made and tested in connection with the design of concrete mixtures, Iowa Highway Commission Series R-48, as shown in Table I. The specimens were cured in damp sand. Also for use with the "Mortar-Void" method, the mortar void data were secured for the sand used. Figure 5 is plotted from the data given in this table.

Design by Water-Cement Ratio Method:

This is a very simple matter as respects quality. If it be assumed that these tests represent the kind of concrete we wish to make, all that is necessary is to take from Fig. 5 the water-cement ratio corresponding to the strength desired. This is all that is needed to control the strength provided the concrete is always plastic. The amount of aggregate used will determine the consistency or workability of the concrete. The most advantageous relative amounts of fine and coarse aggregates and the grading of each are matters of economy not within the scope of this discussion. Since, in this series of tests the consistency was such as would be wanted in the concrete, the data can also be used to give a close indication of the relative amount of aggregate to use; assuming, of course, that the aggregate would be similar in composition and grading to that used in the tests.

As is also shown in Fig. 5, the strength varies with the ratio of aggregate to cement when the consistency is the same. This fact has also been used as a basis for designing mixtures. But since exact methods for control and identification of consistency have not yet been devised, it will be better to use these data merely to estimate the amount of aggregate which will probably give a satisfactory mixture. The proportions should be changed on the job if the degree of workability secured with this mix is not satisfactory.

Problem.—Let us assume the needed strength of the concrete to be 3000 lb. per sq. in. at 28 days. According to the curves in Fig. 5 a water-cement ratio of 0.8 cu. ft. of water per bag of cement is required, and a ratio of 1 lb. of cement to 6.8 lb. of aggregate will probably produce a mixture of suitable workability.

These data can also be made the basis for an accurate estimate of the quantities of materials to be used per unit volume of concrete. The solidity ratio of the test concrete as shown in Table I is 0.865, (i. e., 1–0.135), which should also be close to the solidity ratio of the concrete made with the chosen water ratio and similar materials. The required amount of cement per cubic yard can be computed from the following formula:[1]

$$\text{Bbl. cement per cu. yd.} = \frac{14.1\,d}{1 + \frac{c}{x}a + \frac{c}{y}b}$$

in which d = solidity ratio or percentage of solid matter in a unit volume of concrete,
c = specific gravity of cement,
x = specific gravity of fine aggregate,
a = ratio of fine aggregate to cement by weight,
b = ratio of coarse aggregate to cement by weight, and
y = specific gravity of coarse aggregate.

In the case at hand the total aggregate contains 33 per cent sand and 67 per cent gravel pebbles, both having a specific gravity of 2.66. Hence $d = 0.865$ (from Table I), $c = 3.14$, x (or y) $= 2.66$, $a + b = 6.8$. Substituting in the formula and solving, we find that the amount of cement required is 1.35 bbl. per cu. yd.

A similar estimate can be made from the following formula[2] if it is assumed that the mixing water fully occupies the void space and that no water is lost:

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[1] R. W. Crum, *Service Bulletin*, Iowa Highway Commission, March–April, 1921.

[2] Stanton Walker, *The National Sand and Gravel Bulletin*, September, 1926.

$$\text{Bbl. cement per cu. yd.} = \frac{6.75}{0.5 + x + \dfrac{W_f}{62.5S_f} + \dfrac{W_c}{62.5S_c}}$$

in which x = water-cement ratio, cu. ft. of water per bag of cement,
W_f = pounds of fine aggregate per bag of cement,
W_c = pounds of coarse aggregate per bag of cement, and
S_f and S_c = apparent specific gravities of fine and coarse aggregate, respectively.

For the case at hand solution of this formula gives the required cement to be 1.31 bbl. per cu. yd.

Or, if we assume the volume of concrete to be made up of the sum of the absolute volumes of the water, cement and aggregate, the estimate can be simply made as follows:[1]

For each bag of cement (94 lb.), 0.8 cu. ft. of water and 6.8 × 94 = 639 lb. of aggregate will be used. Assuming the specific gravity of cement at 3.14 and aggregate at 2.66 as in the preceding solutions, we find the contents of such a batch to be:

Water .... 0.800 cu. ft.

Cement $\dfrac{94}{3.14 \times 62.5}$ = ... 0.479 "

Aggregate $\dfrac{639}{2.66 + 62.5}$ = ... 3.85 "

5.120 cu. ft.

Therefore since 5.12 cu. ft. of concrete contains 1 bag of cement, 27.0 cu. ft. will contain $\dfrac{27}{5.12}$ or 5.27 bags or 1.316 bbl. per cu. yd. of concrete.

Design by Mortar-Void Method:

The problem is again to design a mixture that will yield a crushing strength of 3000 lb. per sq. in. at 28 days of age, when made up from the materials represented by the samples used in making the test specimens shown in Table I. In addition to these data the mortar-void characteristics of the sand are needed. These mortar-void characteristics for basic water content and 1.4 relative water content are shown in Fig. 6.

In order to solve the problem by this method, the relation between strength and void-cement ratio for concrete at basic water content for the conditions at hand must be established. Figure 7 shows this relation. The elements of this curve are taken from the test data as follows:

(1) Crushing strength of concrete, age 7 and 28 days, corrected to show on the diagram the probable strength of such concrete at

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[1] F. R. McMillan, *The National Sand and Gravel Bulletin*, November, 1926.

basic water content, instead of the actual strength at the various water contents used;

(2) Crushing strength of mortars, age 7 and 28 days, corrected to show probable strength at basic water content; and

(3) Crushing strength of mortars made up at basic water content.

The corrections for strengths at basic water content are made by means of reduction factors taken from the curve proposed for that purpose by Talbot and Richart, and reproduced herein as Fig. 4. It is interesting to note that the points representative of the three sets of data all agree very closely as to the position of the void-cement-strength curves in Fig. 7.

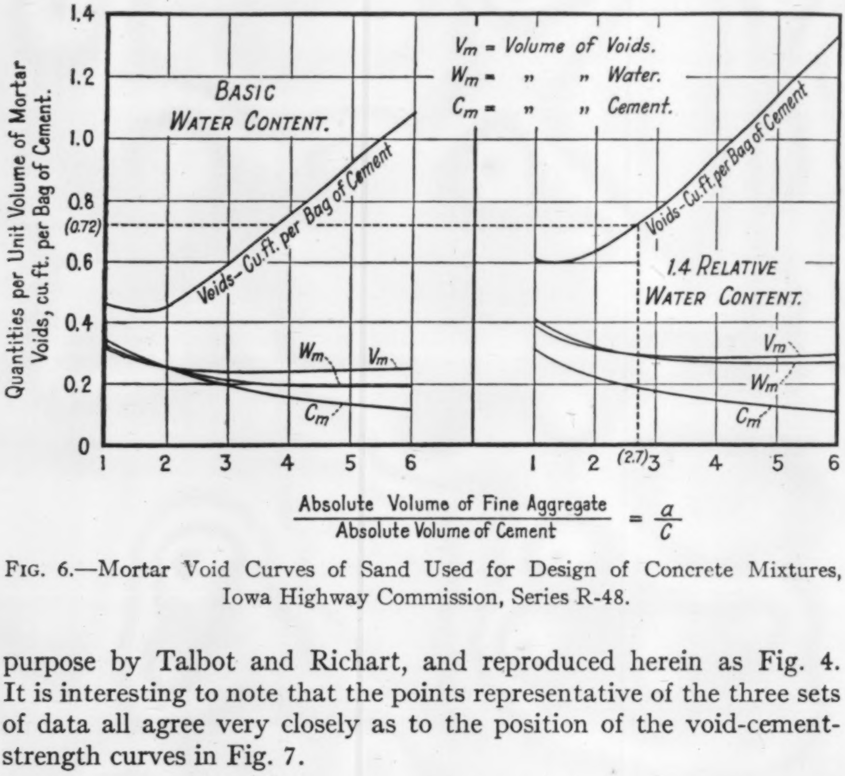

FIG. 6.—Mortar Void Curves of Sand Used for Design of Concrete Mixtures, Iowa Highway Commission, Series R-48.

Problem.—Again assuming the required strength of the concrete to be 3000 lb. per sq. in. at 28 days, it is necessary first to assume the relative water content which will probably give the desired workability. Reference to Table I shows that the concrete of these tests nearest to the strength desired was made with a relative water content of about 1.40, which will therefore be assumed as the basis for the design of the mixture.

From the reduction curve of Fig. 4, we find the strength at 1.4 relative water content to be 0.665 times the strength at basic water content. Therefore our desired strength of 3000 lb. per sq. in. at 1.4 relative water content corresponds to a strength at basic water content of $\frac{3000}{0.665} = 4511$ lb. per sq. in.

From Fig. 7 we find that this strength requires a void-cement ratio of 0.72 cu. ft. per bag of cement. This means that a void-cement ratio of 0.72 should produce 4511 lb. per sq. in. concrete if basic water content is used or 3000 lbs. per sq. in. concrete if 1.4 relative water content is used. Since the latter is to be used, by reference to the mortar-void characteristics of the sand at 1.4 relative water content, Fig. 6, we find that to produce mortar having 0.72 cu. ft. of voids per bag of cement, the ratio of sand to cement in the mortar by absolute volumes will need to be $\frac{a}{c} = 2.70$; the volume of voids per cubic foot of mortar will be $V_m = 0.290$ cu. ft.; the absolute volume of cement per cubic foot of mortar $C_m = 0.190$ cu. ft.; and the volume of water will be $W_m = 0.293$ cu. ft. per cu. ft. of mortar.

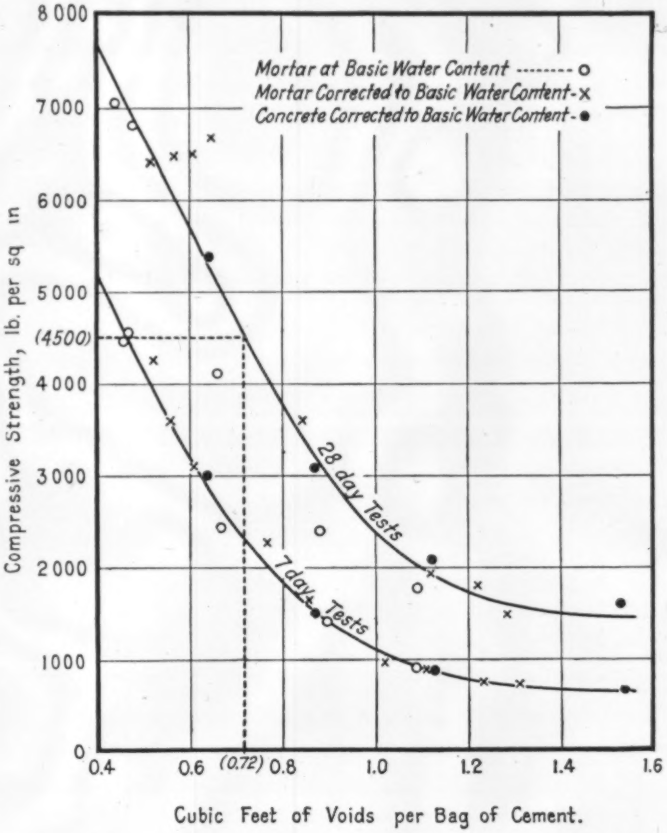

FIG. 7.—Void-Cement Ratio and Compressive Strength of Concrete at Basic Water Content. Tests for Design of Mixtures, Iowa Highway Commission, Series R-48.

$$\frac{\text{absolute volume of sand}}{\text{absolute volume of cement}} = \frac{a_m}{C_m} = 2.70.$$ Therefore, since $C_m = 0.190$,

a_m = 0.513 cu. ft. sand per cu. ft. of mortar. The elements of the mortar will therefore be:

Cement = C_m = 0.190 cu. ft. absolute volume per cu. ft. of concrete.
Voids = V_m = 0.290 " " " " " " "
Water = W_m = 0.293 " " " " " " "
Sand = a_m = 0.513 " " " " " " "

The particular aggregate to be used has twice as much coarse aggregate as fine, both by weight and by absolute volume. Since in this case the specific gravities are the same, there will be 1.026 cu. ft., absolute volume, of coarse aggregate to go with the cubic foot of mortar, and the result will be 2.026 cu. ft. of concrete of the following composition:

Water 0.293 cu. ft.
Cement 0.190 " absolute volume.
Sand 0.513 " " "
Gravel 1.026 " " "

TABLE II.—COMPARISON OF RESULTS.

	Water-Cement Ratio Method	Mortar-Void Method
Water Ratio, cu. ft. per bag	0.80	0.738
Proportions by Weight	1:2.24:4.56	1:2.28:4.57
Solidity Ratio	0.865	0.853
Barrels of Cement per cu. yd	1.35 to 1.31	1.319

These quantities may be reduced to weights as follows:

Cement = 0.190 × 3.14 × 62.4 lb. = 37.23 lb.
Sand = 0.513 × 2.66 × 62.4 " = 85.15 "
Gravel = 1.026 × 2.66 × 62.4 " = 170.30 "

Dividing by the cement factor, the proportions may be stated thus: 1 : 2.28 : 4.57 or 1 : 6.85 by weight.

Volumetric proportions can be easily derived if needed.

The water-cement ratio in this mixture is as follows: 0.293 cu. ft. of water to 37.23 lb. cement or 0.738 cu. ft. of water per bag of cement.

Quantity of Cement Per Cubic Yard of Concrete:

The cement content of the concrete is 37.23 lb. per 2.026 cu. ft. The quantity per cubic yard is therefore $\frac{27}{2.026} \times 37.23$ lb. = 496.1 lb.

496.1 lb. = 1.319 bbl. cement per cu. yd.

COMPARISON OF RESULTS BY TWO METHODS OF DESIGN

Table II gives the comparison of results by these two methods of design.

It should be especially noted that the required quantity of water as determined does not take into account the additional water to be absorbed by the aggregate. An addition for this purpose should be determined by experiment upon the particular materials to be used.

It will be noted from the above discussion that designing mixtures by Talbot's and Richart's mortar-void method leads naturally to arrangement of proportions by weight, since weight is a definite function of the absolute volumes used in solving the problem. On the other hand, loose volumes do not bear any fixed relation to the absolute volumes, and in order to maintain the relationships required by the design, allowances, which are always somewhat indefinite, must be made for the bulking effect of moisture in loose granular material.

EFFECT OF PROPORTIONS UPON EARLY STRENGTH

Recently there has appeared a growing demand for concrete of comparatively high strength at early ages. This demand has been met to some extent by special products, such as the high-alumina cements, by the use of accelerating agents such as calcium chloride, and by the use of ordinary Portland cement under special conditions. This discussion is concerned only with the use of Portland cement.

TABLE III.

From a report "How to Obtain High Early Strength Concrete with Portland Cement" by Portland Cement Association.

Field Mix	Cement, bbl. per cu. yd. of concrete	Slump, in.	Water, U. S. gal. per 94-lb. sack of cement	Water-Cement Ratio, cu. ft. per sack of cement	Time of Mixing, minutes	Compressive Strength at 3 days, lb. per sq. in.
1:2.5:4	1.32	6 to 7	7.8	1.03	1	750
1:2.5:4	1.32	1	6.1	0.82	1	1350
1:1.5:2.5	2.03	1	4.4	0.59	1	2410
1:1.5:2.5	2.03	1	4.4	0.59	5	2860

Examination of the limited data available leads to the opinion that strength at early ages is affected by the proportions of various ingredients, especially cement and water, in the same manner as later strengths. It appears that the methods of design already discussed herein apply to concrete at all ages. Therefore, to design mixtures to yield concrete of a certain strength at three days' age it will be merely necessary to select the water-cement ratio that will produce the required strength, and the amount of aggregates that will produce the necessary workability.

The design should be based upon specific strength data for concrete of the age desired, mixed, cured and stored under the most favorable conditions possible to attain upon the job in question. It has been shown that favorable conditions for high early strength require longer mixing than is usually customary, and plenty of warmth during the fabrication and the curing.

The need for careful control of the entire process should be emphasized. It is commonly recognized that if any chemical action of this nature is to be accelerated, extreme care is necessary to secure good results. The production of high early strength concrete should not be attempted except under adequate expert supervision.

The Portland Cement Association reports the test data given in Table III as indicative of the results that may be secured.

Concrete of comparatively high early strength can easily be produced under normal conditions, if dry and rich mixes are used. The following data in Table IV are from tests made on a paving operation carried on under the Iowa Standard Specifications, without any attempt to produce unusual results. The average compressive strength of cores cut from this pavement at 3 months' age was 4400 lb. per sq. in.

TABLE IV.

Mix by Weight	Cement, bbl. per cu. yd. of concrete	Water, gal. per sack, approx.	Time of Mixing, minutes	Age at Test, days	Number of Specimens	Modulus of Rupture, lb. per sq. in.
1:2.06:2.43	1.74	4.5 to 5.0	1	2	35	340
1:2.06:2.43	1.74	4.5 to 5.0	1	3	6	455
1:2.06:2.43	1.74	4.5 to 5.0	1	4	6	500
1:2.06:2.43	1.74	4.5 to 5.0	1	4	36	540

PART II.—FIELD OPERATIONS

MEASUREMENT OF CEMENT AND WATER

It should be apparent from the discussion on design that no matter how the mixture is designed, maintenance of the proper ratio between cement and water in the field is of paramount importance. Upon this detail depends the average quality of the product. This fact, although recognized, has not been generally applied in practice. Most water measurements are only guesses and are left to the judgment of the mixer operator, under the assumption that if reasonably uniform workability is maintained the desired quality will be secured. Typical of the results under this procedure is the following instance: Special investigations were made upon two such paving jobs, with the rather astonishing result that a variation in water-cement ratio in fresh concrete of from 0.684 to 1.072 cu. ft. of water per bag of cement in one case and from 0.648 to 0.959 in the other, was found. As may be noted from the preceding diagrams, this variation would account for a difference in compressive strength of more than 100 per cent. It seems obvious that this most vital step in the making of concrete should be handled on an accurate basis.

Measurement of Cement:

The measurement of the cement as customarily handled is subject to considerable variation. Some possible sources of error are: the amount in the sack as packed by the manufacturer; the amount left when the sack is emptied; and that blown away by the wind. It is probable that in order to secure the full benefit of accurate water measurement, it will be necessary to weigh the cement.

Measurement of Water:

The maintenance of the correct volume of water per batch is dependent upon three factors: accuracy of measurement; correction for the free water carried by the aggregates; and correction for the water to be absorbed by the aggregate.

Since water is volume constant it can be accurately measured. However, in connection with concrete mixers, the design of the apparatus is complicated by the necessity for rapid measurement and discharge of large quantities. At stationary plants satisfactory accuracy with easy means of adjustment for variable quantity as needed can be secured either by weight or by volume. At moving plants such as are used on highway pavement construction, the problem is more complicated. The chief difficulties are: leakage into the mixer after the discharge valve is closed; crude measurement by means of water gage; and variable effect of pipe-line pressure. At the present time many paving mixers are equipped with water tanks which will furnish a constant quantity of water for a given setting of the adjusting device, under uniform water pressure. Some are designed to be independent of gradient and super-elevation and some require adjustment for these factors. Valves are procurable that are free from leakage for a considerable time. The most common principle used in measuring water at paving mixers is to draw the water from a full tank down to some level which can be controlled from the outside. The common source of error is the filling of the tank. If the pipe line pressure is low, the tank may not fill during the allotted time. With higher pressures, unless the air vents are very carefully designed, air may be trapped and spoil the measurement. Some improvements insuring against these sources of inaccuracy are needed. The cycle of operations upon a modern paving mixer requires that about 42 gallons be measured in 60 seconds and discharged in 10 seconds or less. The inundation method of measuring sand offers good possibilities for accurate measurement of water, especially at stationary plants. The greater portion of the water can be accurately measured with the sand. The remainder to be measured by the usual methods is small enough that the problem is much simplified.

It seems evident from the improvements already made, that water-measuring devices accurate under all usual conditions will soon be developed. It will be necessary, however, for the users to study the equipment and give the manufacturers the benefit of their observations.

Accurate water measurement will not be of much value unless account is taken of the water that comes with the aggregates, and that which is lost by absorption into the stone. In order fully to control water content it is necessary to have the inspector in charge of the proportioning plant equipped with the necessary laboratory outfit for making determinations of these factors and making corrections therefor. The technic for so doing has not as yet been thoroughly developed, but in the writer's opinion it is an imperative necessity. For water control small changes in the moisture content of the aggregates have a relatively large effect upon the result and the drying method for determination of moisture is too slow. The displacement method for determination of moisture in sand and moisture absorbed by aggregate is rapid and, when the technic of the test is developed so that the operator can be sure no air is trapped during the operation, will be accurate and usable. In the report of Committee C-9 on Concrete and Concrete Aggregates for this year (1927) there is proposed for acceptance as tentative a Method of Test for Field Determination of Surface Moisture in Fine Aggregates.[1]

Loss of mixing water through absorption by the aggregate may vary from nothing to a very appreciable amount depending upon the porosity of the material. Probably most materials come to the job already containing practically all of the moisture they will take up, and for such no correction will be found necessary. Certain sandstones and limestones, however, will take in additional moisture. In such cases it will be necessary to determine the additional absorption of the stone for the period of time covering the mixing and placing of the cement, and make the necessary addition to the amount of water to be measured. In most cases this correction would not need to be determined very frequently. It should be remembered that the absorption of such stones from a dried condition will not be of much value in making such corrections. The technic of such tests also needs study and development.

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[1] See *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I, p. 800 (1927).

Adequate water control requires that matters be so arranged that the inspector in charge of the proportioning plant can keep close track of the moisture factors involved, and can notify the inspector in charge at the mixer when and how much to change the tank measurement.

MEASUREMENT OF AGGREGATES

The measurement of sand by loose volume is difficult because of the wide variation in bulk due to moisture and to the manner in which the measure is filled. The volume of sand is approximately constant when dry. Therefore, proportions to mean anything must be based upon a dry condition, and in actual measuring corrections must be made for the expected bulking. Since the bulking also depends upon the manner of filling the measure, this correction will never be better than an approximation. Undoubtedly in the interests of uniformity, the actual volume of material in all batches should be the same, and it would seem that in order to accomplish this, more accurate methods of measurement should be used.

Two methods are available for accurate measurement of sand: inundation and direct weighing. It is known that sand measured under water will yield the same absolute volume of sand grains per unit of volume as dry sand. The inundation method of measurement can therefore be used to give definite quantities of sand and water per batch. The method was first described by R. L. Bertin[1] and has been used on many operations.

Sand can be easily weighed to a high degree of accuracy, and moisture corrections can be easily and accurately made. The equipment cost is practically negligible, and there are no practical difficulties in handling the material.

Neither inundation nor weighing cause delay in production. Coarse aggregates are not subject to the bulking effect of moisture and can therefore be more accurately measured volumetrically. However, greater accuracy is attainable with weights, more definite records of quantities used are secured, and more accurate advance estimates of quantities are possible.

The advantages of accurate measurements of aggregates are: improved uniformity; closer control of quantities of materials; record of materials used; and lower costs.

Uniformity:

One of the most serious criticisms of our concrete work is the lack of uniformity in the quality of the concrete. There are of course a number of factors upon which the uniformity depends. Undoubtedly the accuracy of measurement of the various ingredients is one factor of major importance, and although improvement in this one thing will not entirely solve the problem, the problem cannot be solved until the materials are measured with uniform accuracy.

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[1] R. L. Bertin, "A Method for Measuring Sands in an Inundated Condition," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 404 (1922).

Composition:

The Iowa Highway Commission has for several years studied the composition of the concrete in the roads by making sieve analyses of samples taken once each day from the pavement concrete while yet wet. The cement is removed by washing and the aggregate is dried and screened. Sieve analyses were plotted for each day's run through a paving operation in 1921. According to the specifications the aggregate should have contained 34 per cent of sand, sand being defined as that part passing the No. 4 sieve (0.185-in. square opening). The sieve analyses showed that the sand in the mixes actually ranged from 18 to 50 per cent. In 1924, weighing of aggregates was introduced and sieve analyses plotted in a similar way showed that the sand content for the same specifications ranged from only 30 to 41 per cent. This is not perfect, but it is undoubtedly an improvement.

Cement Content:

One effect of correcting the batches for the moisture present in the aggregates is to reduce the over-run in cement and aggregates due to uncorrected moisture or in other words to make the yield more uniform. The estimated quantities of cement and aggregates are usually based upon dry materials, and therefore in measuring by volume, moisture, especially in the sand, will increase the cement per cubic yard and decrease the yield. In weighing, however, the batch weights can be corrected for the moisture in the aggregates and the yield per barrel of cement kept more constant. The Iowa experience is as follows:

Out of 41 contracts during 1920 to 1923, aggregates measured by volume, the maximum average over-run in cement for a season's work on one job was 10.80 per cent, the minimum was an under-run of 1.8 per cent, and the average over-run for all jobs was 2.00 per cent. Out of 23 contracts during 1924 and 1925, aggregates weighed, the maximum over-run in cement was 3.6 per cent, the minimum 0.3 per cent and the average for all jobs was 1.8 per cent. Variations in slab thickness will always cause variations in recorded cement content per unit of volume, and under rigid inspection the tendency will be to over-run. Data for 1926 are not shown for the reason that on account of the extraordinarily wet season, most of the contracts showed an unusually large over-run in materials, due to great difficulty in building subgrade. It would appear from the above data that some improvement in uniformity was made. These data are presented graphically in Fig. 8.

Material Record:

A valuable result from weighing the materials is an accurate record of the amount of material actually used in the structure. Since such materials are customarily bought and sold by weight, such a record should be of great value, especially to the contractor.

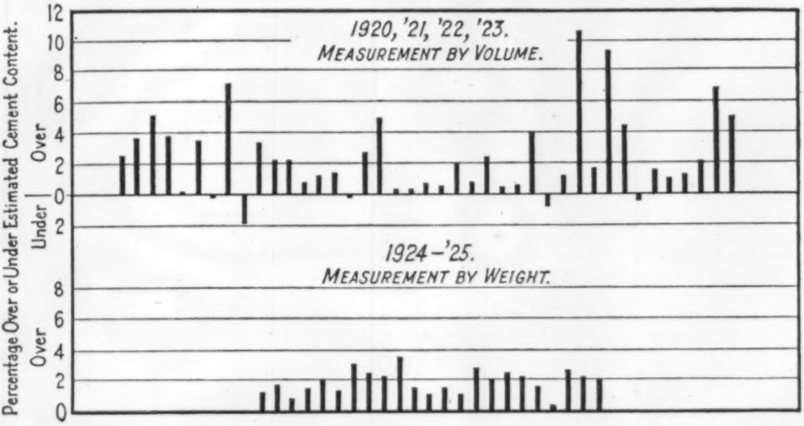

FIG. 8.—Effect of Weighing Aggregates upon Cement Content. Pavement Construction, Iowa Highway Commission.

The length of each line designates the percentage of cement greater or less than the estimated quantity for a season's operation of one mixer crew.

At several times in the past few years, settlements between the Iowa Highway Commission, contractors, material producers, and carriers would have been greatly facilitated had such a record been available. In one case, a paving contractor was billed for sand and gravel and freight at such a rate that every cubic yard of concrete he made would have contained 3900 lb. of sand and gravel. In the proportions specified, this would have meant the production of concrete 105 per cent dense. The producer of the material recognized this absurdity and settled for a more reasonable amount, but this settlement was on a guess and not based on facts as it would have been had the material been weighed when mixed.

Uniform yield and an accurate material record are of great importance to the contractor, and there is no question but that the realization of these advantages will result in closer bidding with consequent reduction in cost to the owner. Any elimination of construction uncertainties is bound to have this effect. The method is therefore justified without reference to the quality of the concrete. However, since it must be admitted that accurate and uniform measurement of materials is the basis for uniform concrete, some improvement in uniformity of concrete by means of weight measurements should result. Examination of the tests of cylinders made from day to day upon a large number of paving jobs indicate this improvement to a limited extent only. The same range of strengths in individual specimens appears in the weighed aggregate jobs as in those in which the aggregates were measured by volume. However, comparison of the average strengths of all of the different jobs on Iowa highways from 1921 to 1926 indicates more uniform results for weighing as respects job averages. These data are shown in Fig. 9. Also the percentage of specimens from the various jobs falling within 20 per cent of the average strength appears to be the more uniform for the weighed aggregate jobs. It would appear from study of these data that there must be some other variation in uniformity of measurement that affects the uniformity of the strength. With the cement and aggregates accounted for, it is obvious that in order to secure the full benefit of the improved methods described, refinement must be made in the control of the water content of the batches.

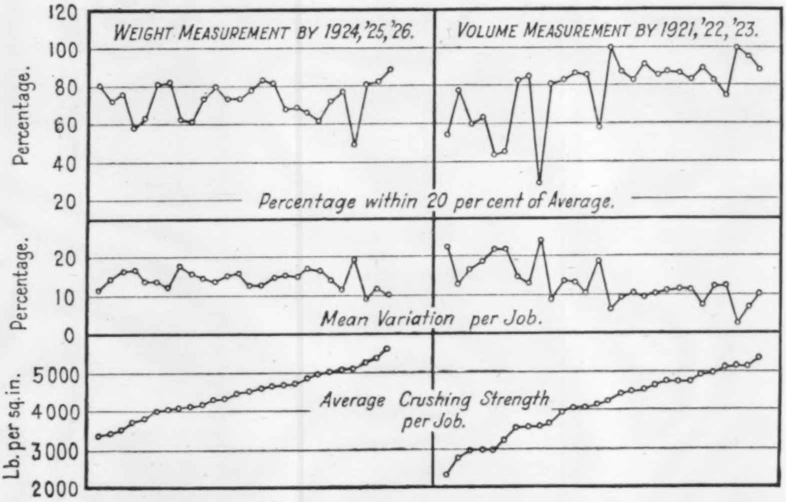

FIG. 9.—Comparative Job Uniformity. Pavement Construction, Iowa Highway Commission.

Each point represents the specimens from a season's operation of one mixer, as made daily.

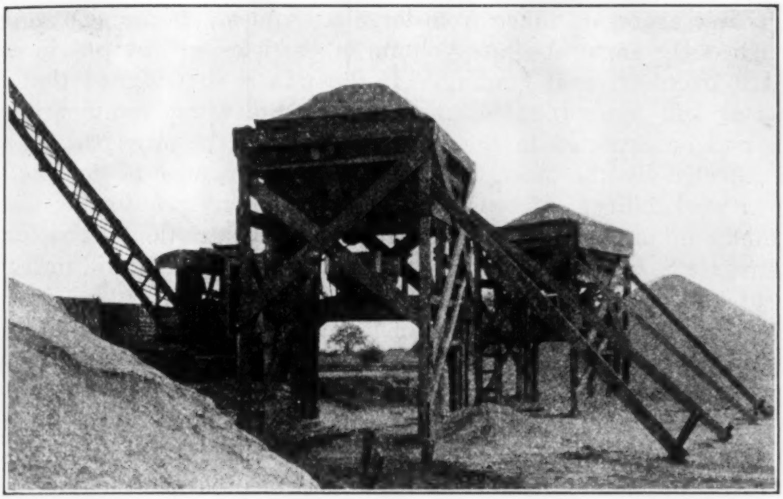

FIG. 10.—A Simple Weighing Arrangement for Loading Trucks.

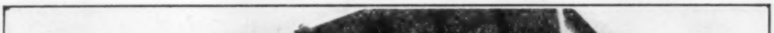

FIG. 11.—Weighing Batcher Plant for Loading Industrial Railway Trains.

Difficulty due to segregation of sizes is sometimes met in the use of coarse aggregate taken from large stockpiles. If the aggregate is weighed the same absolute volume of particles will be put in each batch irrespective of grading. If the mix is so designed that the mortar will more than fill the voids in the worst combination of sizes to be expected in the coarse aggregate, the strength will not be affected by the stockpile variation. There may be some effect upon workability. If considerations of economy require the use of a minimum proportion of mortar, the stockpile variation of the coarse aggregate may cause trouble. In such a case volumetric measurement of the coarse aggregate would roughly compensate for the effect of segregation, for with an increase in voids, less total stone would go into the batch. The advantage of definite batch composition and yield would be lost in this case.

A much more definite method would be to supervise the building of the stockpile so that segregation could be kept within allowable limits. Upon important work it is possible that it may be found worth while to separate the coarse aggregate into several sizes. A complete discussion of this problem would involve considerations of economy rather than quality and is, therefore, without the scope of this paper.

Apparatus:

Several arrangements of weighing apparatus have been successfully used. Simple field-built bins and hoppers with ordinary platform or mill scales have been used, as well as permanent equipment designed and built by manufacturers of paving equipment.

Four general types are in use as follows:

1. Bottom dumping hopper on lever weighing system with direct reading dial scale.
2. Batcher device adapted to weighing system, usually with pipe lever scales.
3. Bottom dumping hopper on platform scales.
4. Side dumping hopper on platform scales.

In the experience of the Iowa Highway Commission those installations have been found most satisfactory, which are so arranged that the scale beam and operator are at the same elevation as the weighing hopper and that the operator can observe the inside of the hopper and easily remove excess material if necessary.

Figure 10 illustrates the simplest type of installation for loading trucks and Fig. 11 illustrates the use of a battery of four suspended hoppers for loading industrial railway trains.

MIXING OF CONCRETE

By Duff A. Abrams[1]

Synopsis

The paper discusses the mixing of concrete from the point of view of its influence upon the quality. The principal classes of mixers are described and their advantages and limitations pointed out.

The effect of time of mixing concrete is discussed, with reference to reports of several investigations that have been made on this feature. Modern requirements for mixing concrete for general purposes and for concrete road construction are quoted from technical specifications.

Concrete may be defined as an intimate mixture of cement, water and aggregates which is permitted to set and harden under favorable conditions. The properties of concrete are determined largely by the qualities of the constituent materials and the proportions in which they are used. This paper deals solely with mixing, hence, it is assumed that the selection and proportioning of materials has been properly handled.

The function of mixing concrete is two-fold: (1) Bringing the water in contact with the cement, and (2) forming a combination as homogeneous as practicable of the cement and aggregates. These two operations may be performed simultaneously or separately. In certain instances the concrete materials are mixed dry and put into place, the water being added later.

Earlier mixing of concrete was done by hand with hoes or shovels. This method of mixing was almost universally followed up to about 1870, when machine mixers began to make their appearance. During the past twenty years, concrete mixers have been greatly improved and machine mixing has become almost universal. Hand mixing is used only on unimportant work or in isolated locations.

Types of Mixers

Concrete mixers can be divided into the following three classes:

(1) Rotating drum with fixed blades or buckets;
(2) Rotating blades, stationary drum; and
(3) Gravity.

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[1] Director of Research, International Cement Corporation, New York City.

The rotating drum mixer with fixed blades or buckets for lifting and distributing the concrete during mixing is typical of modern practice for construction and paving work.

Rotating blade mixers are generally used in concrete products plants where semi-dry concrete is required.

Gravity mixers are generally considered unsatisfactory and are seldom used except where conditions such as ample head room make such a mixer feasible.

A "truck-mixer," consisting of a special truck body provided with a rotating blade mixer, has recently been placed on the market. The dry concrete materials are loaded in the body-mixer at a central proportioning plant; the materials are hauled dry to (or nearly to) the work; two or three minutes before dumping the batch the water is admitted from tanks carried by the truck and the mixing started. The mixer is driven by the truck motor. The entire batch is then dumped through an end gate.

Standardization of Mixer Sizes.—The Mixer Manufacturers Bureau of the Associated General Contractors of America have standardized their practice with reference to sizes and certain other features of mixers for construction and paving work. The following are the size designations used for a 1: 2: 4 concrete mix:

Size of Batch in Bags of Cement for 1: 2: 4 Mix	Standard Sizes of Mixers, Capacity in Cubic Feet of Mixed Concrete	
	Construction (S)	Paving (E)
½	3½	..
1	5 and 7	..
2	10	10
3	14	13
4	21	..
6	28	27

The sizes of batch given above will be changed for concrete of other proportions. Mixers of two cubic yards capacity or larger are being used, but these are considered special.

Construction and paving mixers are generally equipped with self-contained power units which are operated by gas or steam engines or by electric motors. Paving mixers are provided with loading skip and with boom and bucket for distributing concrete on the subgrade and are self-propelling at speeds ranging from 0.5 to 1.8 miles per hour.

Water measuring tanks are a part of standard equipment of the larger mixers for construction and paving work. A siphon overflow permits the tank to be filled to the required depth each time. The water is discharged through a pipe of large capacity which is fitted with a quick-acting valve.

Probably the most difficult problem before the mixer manufacturer is the accurate control of the quantity of mixing water which goes into each batch. The difficulty is increased by the presence of varying amounts of moisture in the aggregate and by the necessity for quick action. The effort to attain accurate measurement of water is an evidence of the general recognition of the importance of quantity of mixing water in concrete.

Time locks are available which lock the discharge lever until the drum has made the number of revolutions for which the apparatus is set. The use of such a lock prevents the premature discharge of the batch and also, by sounding a gong, calls the attention of the operator when the time for discharging the batch has arrived.

EFFECT OF TIME OF MIXING CONCRETE

A number of studies have been made of the effect of time of mixing concrete on its strength and other properties. The following reports may be referred to for more detailed information:

"Effect of Time of Mixing on Strength and Wear of Concrete," by D. A. Abrams, *Proceedings*, Am. Concrete Inst., 1918.

"Tests of Concrete Mixer," by W. K. Hatt, *Proceedings*, Am. Concrete Inst., 1921.

"Concrete Mixing Tests," by C. S. Pope, *California Highways*, February, 1926.

The tests show that the strength of concrete of all mixes and consistencies is increased as the time of mixing is prolonged, within the range of mixing times used. The rate of increase is quite pronounced during the interval of 15 to 30 seconds, but lessens as mixing is prolonged. Within the range of mixing times of 15 seconds to 10 minutes the 28-day strength of concrete was increased about 10 per cent by doubling the time of mixing. In other words, concrete mixed for 2 minutes was 10 per cent stronger than similar concrete mixed 1 minute; mixing 1½ minutes gave strength about 6 per cent greater than 1 minute. It seems proper to conclude that there is little advantage in mixing concrete longer than 1 minute after all materials are in the drum.

SPECIFICATIONS

Joint Committee Specifications.—The following paragraph from the 1924 Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete,[1] give the modern requirements for mixing concrete for general purposes:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 303 (1924).

"*Machine Mixing:* The mixing of concrete, unless otherwise authorized by the engineer, shall be done in a batch mixer of approved type which will insure a uniform distribution of the materials throughout the mass, so that the mixture is uniform in color and homogeneous. The mixer shall be equipped with suitable charging hopper, water storage, and a water-measuring device controlled from a case which can be kept locked and so constructed that the water can be discharged only while the mixer is being charged. It shall also be equipped with an attachment for automatically locking the discharge lever until the batch has been mixed the required time after all materials are in the mixer. The entire contents of the drum shall be discharged before recharging. The mixer shall be cleaned at frequent intervals while in use. The volume of the mixed material per batch shall not exceed the manufacturer's rated capacity of the mixer.

"The mixing of each batch shall continue not less than one minute after all the materials are in the mixer, during which time the mixer shall rotate at a peripheral speed of about 200 ft. per minute."

Typical Highway Specifications.—Below is an extract from the 1926 Specifications of the New York State Highway Department; it is typical of recent practice in concrete road construction:

"No concrete shall be mixed while the air temperature is at, or lower than 40° F. and no materials containing frost shall be used. Cement or fine aggregates containing lumps or crusts of hardened materials shall not be used. The concrete shall be mixed only in such quantity as is required for immediate use and any which has developed initial set or has been mixed longer than 45 minutes shall not be used. No concrete is to be placed on a frozen subgrade.

"All equipment necessary for the laying of pavement must be on hand and must be inspected and approved before concreting operations are begun by the contractor. Concrete shall be mixed thoroughly in a batch mixer of an approved type and capacity for a period of not less than one minute after all material is in the drum and during this period the drum shall make not less than 12 nor more than 18 revolutions per minute.

"The entire contents shall be removed from the drums before materials are placed therein for the succeeding batch. The mixer shall be equipped with an approved boom and bucket discharging device."

Central mixing plants for concrete have been established in a number of cities. The concrete is mixed in a permanent proportioning and mixing plant and dumped from the mixer into trucks which haul it to the work. The following paragraph bearing on this subject is found in the 1927 Specifications of the New Hampshire State Highway Department:

"The use of a central mixing plant and the transportation of mixed concrete to the road will be permitted, provided there is no unreasonable segregation of the concrete when it arrives at the point where it is to be deposited on the road, and provided further that the period of time elapsing from the time the concrete is mixed until it is deposited on the road does not exceed 30 minutes. The concrete shall be thoroughly rehandled with shovels or other suitable means, if necessary, to obtain a uniform consistency before tamping."

Critical studies of the performance of concrete mixers have been carried out during the past two years by the U. S. Bureau of Public Roads. These studies were made on concrete road construction jobs in several different states. Certain phases of this work are covered in detailed reports by J. L. Harrison, published in *Public Roads* during 1926. The results of their studies of time of mixing have not been published.

CONVEYING AND PLACING CONCRETE

By Nelson L. Doe[1]

Synopsis

The paper discusses certain features of conveying and placing concrete and their effect upon the quality of concrete. The several operations involved in the distribution of concrete are presented and the strong and weak points of each described.

In this way are discussed mixer discharge, conveying concrete by hoisting, chuting, wheeling, trucking, belt conveying, and pneumatic placing. The effect of hydrated lime and celite on conveying and placing mixtures is discussed.

Within the past five years, much study has been given to the character and proportions of the raw materials from which concrete is produced, so that now concrete of a predetermined strength can be designed. The distribution of concrete for the ordinary structure consists of several operations. These will be discussed in sequence, starting with the discharge of the concrete from the mixer, and following through the operations of hoisting, chuting, wheeling, trucking or conveying by means of belt conveyors, an attempt being made in each case to bring out the strong and weak points in the method of distribution described. Attention will also be given to the use of admixtures such as hydrated lime or celite and their effect during the transportation and placing of the concrete in the forms.

Mixer Discharge

There are two common forms of mixers in general use: the mixer which is discharged by means of a chute, pivoted so as to swing into the center of the drum; and the tilting drum type. The former is by far the more universally used on ordinary contract work. In selecting a mixer of this type, care should be taken to see that the chute when in discharging position has pitch enough so that concrete of a stiff consistency will slide along it without accumulating to block the opening from the drum, otherwise the tendency is to add water to the mix until it can be discharged without difficulty. The discharge should be so arranged that the operator can inspect the batch without tilting the chute at such an angle that the weight of the outpouring batch prevents the returning of the chute to the normal running position, in case the batch requires a slight change in consistency. The chute should be so designed that the mixer can be emptied quickly, for when a large volume of concrete is carried there is less tendency for it to separate. Chutes should extend well into the center of the drum, past the line of dippers; otherwise the batch will be discharged with either the heavy aggregate first, or last, or it may not be discharged completely.

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[1] Turner Construction Co., New York City.

Concrete having considerably stiffer consistency can be discharged from a tilting mixer, in less time than from the chute-discharging type. This is a distinct advantage and is one of the chief attractions of the tilting machine.

The discharge from either type of mixer should be arranged so that the concrete will not travel any considerable distance nor spread out across any sloping surface. The discharged concrete should not slide down the side of the receiving box or bucket. It should be poured into it, striking near the center and with approximately a vertical fall. If this action takes place, there will be little segregation of aggregates.

CONVEYING CONCRETE

Hoisting:

Buckets for hoisting concrete are of three general types: (1) the large-top, rather shallow, cylindrical-bottom type; (2) the deep, squarer, rolling-discharge type; and (3) the pyramid bottom-discharge type bucket, as now usually specified for use with steel masts. The deeper bucket offers less opportunity for the spreading out of the concrete, as its cross-section is not so large for given capacities. It also possesses an advantage in dumping, as the pouring action is greater than with the shallow bucket. The load is of much greater thickness when discharged from the deeper bucket than when it slides out of the shallow one, and it is discharged quicker. Neither bucket should be dumped into a hopper that has a large bottom. The concrete should be poured into itself so that the surface of the mass rises uniformly around the entering stream.

A bucket which discharges through a gate at the bottom causes much less segregation of aggregates than the buckets which unload by dumping the concrete over the edge. This is a decided point in favor of the pyramidal type of bucket. A batch of concrete of ordinary consistency improperly chuted from the mixer into a bucket of this type with noticeable segregation will be discharged through the gate in the bottom partially remixed. If interest in controlling the quality of concrete continues to increase, it would not be surprising to see the top-dumping bucket disappear entirely in favor of the bucket which dumps through a bottom gate.

Hoppers so designed that a swirling action or eddying of the mass is noticeable should be corrected, for whenever this takes place the aggregates tend to separate. Gates should shut tight and quickly, otherwise rich material wastes away, leaving coarse aggregates against the gate.

Although the actual hoisting of concrete in buckets has little effect on quality, improper loading or unloading of the bucket will cause segregation, and these features should be carefully arranged. The less concrete is handled the less segregation and the more uniformity in the deposited concrete. The moving of concrete a few feet in an improper manner may affect its quality more than properly conveying it long distances.

Chuting Directly from Mixer or Distributing Hopper:

Possibly in the past, chuting operations have justly deserved some of the adverse comment they have received. With modern ideas of water control and consistency, most of the objections to chuting concrete from the standpoint of quality have been automatically removed. Concrete of proper consistency can readily be chuted. Concrete of a very wet consistency cannot be chuted without marked deterioration. These facts are a strong argument in favor of chuting concrete, for chuting furnishes a check on the consistency of the concrete. With given materials, the limiting angles of distributing chutes can readily be ascertained after the action of a few batches has been studied, and once the chutes are erected accordingly, little segregation is experienced.

Different aggregates and chutes produce different effects on the velocity of the concrete carried, so these limiting angles vary considerably. Gravel concrete will be best handled at a slope of approximately one vertical to three horizontal, while concrete of the same consistency made with broken stone or slag may require a slope of nearly one to two.

Details of chutes have much to do with the flow of concrete and improvements in chute designs have steadily been made. The increased size of end connections and their improved shapes have done much to eliminate overflowing and clogging. This permits a considerably stiffer concrete to be carried safely. More harmful effects may result in chute distribution due to improper discharge at one point along the line than in the entire line of chutes properly adjusted. A proper receiving hopper should be provided at the end of the chutes; or if the concrete is placed directly in the forms, it must not fall freely more than a few feet, or be shot out at an angle. Long runs of chute should always discharge into a hopper having a bottom gate.

Conveying with Two-Wheeled Carryalls:

From a practical point of view there is little to criticize regarding this method, although study shows that concrete transported by carryalls segregates *en route*. Due to the different specific gravities of the materials and the vibration of wheeling, all heavy aggregates tend to settle while the fine material and excess water rises to the top of the carryall. In batches of wet consistency, segregation is more pronounced than in batches of ordinary consistency.

Tests indicate that concrete placed in the forms by wheeling in carryalls will be segregated to such an extent that variations from the mean strength of 20 per cent will not be unusual. The redeeming feature of this, however, is the fact that each carryall contains less than six cubic feet of concrete, hence no great volume is affected. As each carryall is placed in the forms the opportunities for remixing are present, so that while segregation during transit may help to explain non-uniformity of field tests, the strength of the concrete over any considerable area is probably quite uniform.

Concrete discharged directly from the mixer to carryalls eliminates all hazard of segregation due to chuting or hoisting, but it is not easy to fill carryalls properly from the mixer chute. The concrete leaves the mixer too rapidly, and strikes the carryall at too flat an angle, causing it to swirl and load the carryall unevenly. There is a tendency to load carryalls when they come to the mixer regardless of whether the batch has been mixed sufficiently, which is very detrimental to quality.

Distribution by Industrial Cars or Derricks:

For mass work, the dumping of concrete directly from the mixer into derrick-handled buckets is most satisfactory. Buckets can be set so that the mixer discharge strikes the center of the bucket practically vertically. There is little vibration as the bucket is hoisted or lowered to the position where the concrete is to be deposited.

Concrete discharged directly from the mixer to industrial cars shows no unusual segregation of aggregates, provided the discharged concrete drops vertically into the body. There is always less segregation in cars having deep bodies than shallow ones. For the best results, cars and buckets should be equipped with suitable dumping gates, through which the velocity of flow is controlled. The settling of heavy aggregates which may occur during transit is largely counteracted by the fact that the finer aggregates from the top tend to flow more rapidly when the bottom gate is opened than the heavier closely packed aggregate near the bottom. The result is a very satisfactory remixing. With cars or buckets that dump over the top this process is reversed; the fine material which has risen during transit is discharged first over the top, leaving the hard-packed aggregates with no chance of remixing, as the fine materials flow much faster than the heavy aggregate if separated. The importance of bottom-dumping equipment cannot be over emphasized.

The track for industrial cars should be free from abrupt turns or irregularities which transmit jolting action to the concrete.

Distribution by Motor Truck:

Motor-truck distribution causes segregation of concrete while it is in transit, more than while it is being loaded or unloaded. This is due to the vibration that the concrete receives. Distribution by the ordinary open-top truck body is impractical because of the fact that when the concrete is discharged from the truck by elevating the forward end of the body, the fine materials, together with the excess water, are discharged first, followed by the heavy aggregate. Concrete handled in this manner requires complete remixing. Delays, if encountered, cause the concrete to become so firmly attached to the truck body that these heavier aggregates must be loosened with a pick. Trucks designed to carry a body shaped much like a concrete hoisting bucket, with discharge gates at the bottom, have given more efficient results, reducing to a considerable extent the segregation during transit and assisting in the remixing of the concrete when it is discharged.

An enterprising distributor of ready-mixed concrete in San Francisco has constructed on his trucks a long cylindrical mixing body into which dry materials for three yards of concrete of given specifications are placed. Water for the batch is carried in a separate compartment on the truck. Upon arrival at the receiving hopper, the power from the truck engine is thrown into the mixing blades, the water added to the dry batch and freshly mixed concrete of any desired consistency is discharged from the truck. This development seems to be a step forward in the handling of concrete by truck, since it eliminates segregation during transit and removes dangers due to delays in transit.

Distribution by Belt Conveyors:

Transportation by means of belt conveyors has been successfully used on core walls of dams and other works where sufficient volume of concrete warranted belt installations.

For well-mixed concrete having a slump of not over 5 in. practically no segregation of aggregates takes place on belts even on considerable inclines. Concrete of a sloppy consistency cannot be handled by inclined belts without marked segregation, as the fine materials tend to flow down the center of the incline leaving the larger aggregates on the edges of the belt. Proper loading and unloading arrangements for belt conveyors are important. Concrete conveyed by belts should be discharged into a receiving hopper of ample capacity, so that the concrete from the belt falls directly into a mass of concrete, and does not strike a sloping surface. Belt conveyors should be charged from a hopper through a bottom gate and not fed directly from a mixer chute. In this way the discharge can be gaged so that a continuous stream of concrete can be maintained on the belt with a single mixer, giving extremely uniform conditions of distribution.

Distribution by Pneumatic Methods:

On practically all tunnel work, placing concrete in the forms by means of pneumatic machines has been found most satisfactory and efficient.

In pneumatic placing the discharge end presents the greatest liability for segregation of the aggregates. If the discharge line is simply extended into the section of tunnel lining to be concreted, the force of the air sends the heavy aggregate to the opposite end of the form, while the fine materials fall in a sharper curve with disastrous results. The discharge pipe should be carried to within a few feet of the far end of the form and be properly baffled. As filling progresses, short lengths of the discharge pipe are removed, until only a small space at the near end of the form remains open. This space is left until the next fill is made, forming a key between the sections. From a quality standpoint, pneumatic placing has everything in its favor. Inspection of tunnels shows that air-placed concrete is much freer from honeycombing than hand-placed concrete. Leakage through concrete placed by air is also much less.

EFFECT OF ADMIXTURES ON CONVEYING AND PLACING CONCRETE

The use of admixtures is now steadily gaining favor with those familiar with placing concrete. Authorities agree that changes in the final strength of concrete containing suitable proportions of proper admixtures will not vary much more than the percentage of the admixture used.

The real value of admixtures does not lie in added strength given to the concrete, but in increased workability. This is especially true with the leaner mixes. The larger aggregates in concrete containing admixtures seem to be coated more completely with a covering of fine material which clings to the aggregates, decreasing their rolling action but increasing their sliding qualities.

Rich stiff concrete possesses workability enough ordinarily so that no improvement for proper handling is required.

Hydrated lime has been added to concrete on various classes of work for years and no detrimental effects have been found. Celite is a comparatively new material, and its effects on such qualities of concrete as strength, absorption of moisture, weathering, etc., are still under observation.

For many purposes either material can be used as an admixture without question: for example, for fireproofing around structural members; for increasing the workability of harsh mixtures where the aggregates cannot easily be varied; where complicated form work or reinforcing is encountered; where concrete is to be placed on sloping forms; or where the workability of the concrete must be retained for a considerable time while some other portion of the forms is being filled. On other classes of work, such as exterior walls exposed to severe weather conditions, concrete designed to resist water pressure, or concrete requiring especial strength, probably either hydrated lime or additional portland cement would produce the best results.

PLACING CONCRETE IN THE FORMS

The quality of concrete can be damaged considerably by careless placing in the forms. One of the fundamentals of placing is that concrete falling through space tends to segregate. Another is that the surface of flowing concrete must be kept approximately level. If these two points are well guarded, and care used in working the concrete against the form, and between the reinforcement, well-placed concrete will be the result.

While cleaning of forms may appear too commonplace for consideration, it is nevertheless a factor in the proper placing of concrete and needs emphasis. The bearing value of columns or walls is decreased in proportion to the amount of refuse left in the forms.

Form details affect the appearance and quality of concrete, also. No large cracks between boards or panels can exist without draining some of the richest material from the concrete. Open spaces between concrete poured, and forms about to be filled, cause most of the honeycombing seen near joints between fills. Often some portion of a structure calls for a richer mix than usual. In such cases the foreman should be supplied with a location plan showing the special portion marked distinctly and he should be checked up on the mix at the time of pouring. On difficult work it is not unreasonable to distribute the placing of vertical fills such as high columns or walls over an entire day, allowing generous settling periods between the pourings. Concrete should never be dumped onto the floor forms adjacent to a column or wall to flow over the edge into the form, but should be poured into a portable box having a 6-in. opening directly over the center of the column or wall.

When columns have spiral reinforcement, it is especially important to deposit the concrete inside of the spiral. When pouring such columns at least three wooden rods should be outside of the spiral, to insure the proper centering of it. Similar rods inside should be worked at intervals, to assist in compacting the concrete. It is safer and more satisfactory to fill supporting columns and floor on the same day, for the laitance which rises in the columns continues to rise to the top of the floor concrete. Rapping of the forms gently is of considerable assistance in increasing the flow of concrete around reinforcement and into corners. Continued vibration is not necessary on ordinary work, but is a valuable feature which must not be overlooked on complicated form work.

No excess water should accumulate on the floor panels in front of a floor pour, but the material should be kept workable enough to fill the forms completely. The practice of wheeling over concrete poured one or two days previous should be discouraged, especially during cold weather.

Far too little attention is paid to necessary clearance between the steel and the forms on most operations, although it is well known that steel left too close to the surface will in a short time force very sizeable pieces of concrete from the face of the structure, greatly affecting the appearance and ultimately the strength. While placing concrete, this point should receive the constant attention of the entire concrete gang, as well as the whole attention of at least one man assigned for the special purpose of keeping all steel back from exterior surfaces.

When considering methods to be used for handling concrete, it is well to remember that a thoroughly reliable and suitable set-up for distribution under one set of conditions may prove entirely out of place in different circumstances. In the selection and operation of all concrete-handling equipment, theoretical layouts and devices must be accepted only after they have been tested and found to work out to advantage under service conditions.

CONSTRUCTION JOINTS AND EXPANSION JOINTS

By R. L. Bertin[1]

Synopsis

The paper defines construction and expansion joints and discusses their effect upon concrete structures.

The harmful effect of laitance at horizontal joints is discussed and careful control of the plasticity of concrete is recommended to prevent the accumulation of such laitance. Provisions for securing water tightness of horizontal joints are suggested.

The influence of vertical construction joints of reinforced-concrete members subjected to bending is pointed out and the proper location of such joints in slabs, beams and girders is indicated. In structures intended to be water tight, special provisions must be made to make vertical construction joints tight and some methods are suggested.

The difference in function between expansion and construction joints is emphasized and the elements affecting the location and frequency of expansion joints in concrete structures are enumerated.

Joints in concrete structures are generally divided into two classes: (1) construction joints and (2) expansion joints.

A construction joint is a joint introduced whenever conditions in the field make it necessary to stop pouring concrete and resume at a later date. The field conditions causing an interruption in the pouring operations are numerous, many of them accidental, so that the location of construction joints cannot be predetermined with any degree of certainty. Among them may be mentioned the capacity of the concreting plant, forced stoppage due to shortage of materials, breakdown of machinery, sudden storms, etc.

An expansion joint is one introduced in the structure to meet requirements of design. The general purpose of such joints is to relieve the structure of excessive internal strains due to volumetric changes caused by temperature and moisture variations in the concrete.

Construction Joints

There are two types of construction joints, horizontal and vertical. Horizontal joints occur in columns, walls, dams and the like, and vertical joints in walls, beams, girders and slabs.

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[1] Chief Engineer, White Construction Co., Inc., New York City.

Any joint in a concrete structure destroys the monolithic property of the structure at that plane, and, since construction joints may occur almost anywhere in the structure, rules governing their location and construction must be promulgated to minimize the harm being done to the structure.

Concrete possesses the property of expanding and contracting under the influence of moisture and heat. As it dries out after being placed, a progressive shrinkage of the mass takes place, reducing its linear dimensions. The mass, when set, is further affected volumetrically by changes of temperature. These volumetric changes cause relative motion between the edges of adjoining concrete masses cast at different periods, and special provisions must be made at these joints to correct for the effects on the structure, resulting from such motion.

Horizontal Construction Joints:

Plain concrete members having horizontal construction joints are not affected materially by any volumetric change, for the reason that the upper section usually remains in contact with the lower. If, however, the upper part is restrained, the lower part may shrink away from it and leave a space between the two sections which, under certain conditions, may prove detrimental. Where the concrete mass is reinforced with steel bars crossing the construction joint, the bars, if of sufficient magnitude, may prevent the sections from keeping in contact, thus leaving a space between them.

Horizontal joints through such structures, intended to resist the elements or water pressure, should be constructed in such a way that tightness will be maintained, even if the sections separate. This may be accomplished by means of a tongue and groove or ship-lap joint or a metal strip embedded in both sections.

Whenever concrete is cast in vertical members, unless the concrete is very carefully controlled in its plasticity, water and the finer particles in the concrete will accumulate at the top. This scum or laitance, as it dries, forms a chalky mass of little or no strength, very porous, and susceptible to disintegration if subjected to moisture. If, therefore, a vertical concrete structure is poured in parts and the laitance allowed to remain, the seam thus formed weakens the finished structure. It is, therefore, imperative that all trace of scum be removed from the top of each cast, but, better still, that the concrete be controlled so as to prevent the accumulation of scum at the top. This formation of scum is a form of segregation due to several causes: Improper grading of the materials, defective proportions, and excessive quantity of water used in the mix. The practice is sometimes resorted to of absorbing this scum by means of a very dry mixture being used at the top of the cast. This method is not to be recommended for the reason that this scum is not structurally sound and, when present in large quantity, will not produce concrete of good quality when mixed with the dry mixture. The best practice is, therefore, to prevent the formation of laitance and, if it is present, to remove all trace of it while still fluid.

Vertical Construction Joints:

Vertical joints in plain concrete structures fall under the classification of expansion joints, for their very existence simply divides the structure into independent units. When, however, vertical joints occur in reinforced-concrete members, the member, although separated, still remains, as a whole, a complete member, and the influence of this break in the continuity of the concrete on its performance is more involved. A reinforced-concrete member subjected to bending, be it a slab, a beam, or a girder, is resisting stresses of varying magnitude and direction. The general assumptions of design are that the concrete resists, wholly or in part, the compressive stresses and the reinforcing steel all tensile stresses induced in the structure by external forces. Whenever a construction joint is introduced in such a member, thereby severing the concrete, the compressive stresses which the concrete is called upon to resist must cross the plane of cleavage and this plane should, therefore, be normal to the direction of the stresses. Other stresses induced in a reinforced-concrete member, subjected to flexure, are horizontal and vertical shear. These stresses are partly resisted by the concrete and any plane of cleavage destroys the resistance of the concrete to shear. Therefore, it follows that horizontal construction joints should be avoided in any concrete member subjected to flexure, and that any vertical construction joints must be located where the vertical shear is nil, which is that point where the bending moment reaches a maximum. In general, this location is at or near the center of the spans, except in the case of members carrying eccentric concentrated loads where special investigation should be made to determine the proper location if a construction joint is necessary.

The faces of such joints should be cast against a formed stop and the concrete properly puddled against it to insure a true face of sound concrete. Under no circumstances should the concrete be allowed to slope at a natural angle and concreting resumed, if a period greater than two to three hours is to lapse between casts.

Where vertical construction joints occur in structures intended to be water tight, special provisions must be made to make such joints tight. This may be done in several ways, the most common method being to make the joint Z shape and covering the old surface with a coating of alkali-resistant waterproofing elastic medium before applying fresh concrete against it. Another method is to embed a metal strip across the joint, crimped at the joint, thus providing for slight motion without causing any strain in the strip of metal or the concrete in which it is embedded. When a cement finish is applied to the structure, a blind or open joint in the finish coat should be provided directly over the construction joint, thereby localizing the crack which is likely to develop over the construction joint.

EXPANSION JOINTS

An expansion joint differs from a construction joint in so far as the expansion joint is located in a structure at certain intervals to isolate one part of the structure from another, each part being considered complete in itself and independent of the adjoining one. The purpose of dividing a structure into units is to limit the effects of volumetric changes on the structure as a whole. The radical difference between a construction joint and an expansion joint is that with the former, internal stresses pass from one side of the joint to the other, whereas with the latter, the internal stresses terminate on one side and start again on the other.

It follows, therefore, that no material offering any resistance should cross from one side to the other of an expansion joint, and perfect freedom of motion should be allowed between adjoining sections.

A diversity of opinion exists in the minds of engineers as to how large a unit may be built without expansion joints. Computation may be resorted to in determining the expansion and contraction of structures caused by a given range of temperature. The time of the year when the structure is built has an important bearing on such computations. The shrinkage of concrete which takes place during the early setting periods is also a large factor. This shrinkage varies in magnitude with different grades of concrete. The exposure of concrete to dry and wet conditions also causes volumetric changes in the mass, so that accurate determination of location of expansion joints predicated upon so many varying factors are nearly impossible. Research work along this line is needed and would make the subject of expansion joints a more exact science.

Expansion joints in concrete roads are a matter which has been and is being given a great deal of study and, as yet, no definite rule has been evolved as to what is the best practice.

The purpose of formed expansion joints is essentially to prevent the structure from forming its own expansion joints in the form of cracks which might weaken the structure or destroy its usefulness, as in the case of reservoirs, tanks or any form of fluid-tight structures.

In locating expansion joints in a structure, due consideration must be given to the resistance of the structure as a whole, for contraction will naturally cause a structure to crack through its weakest plane, and, therefore, expansion joints should be provided at places where the structure is weakened by a reduction in section.

It is not always possible to construct expansion joints so that the adjoining parts will be entirely independent of each other; for instance in buildings it is sometimes necessary to have one part lap over and bear on the other. In such cases, a sliding seat should be provided which will offer as little resistance to sliding as possible. This type of joint is to be used only when a through joint is absolutely impossible and then steel plates or roller bearing on an ample seat should be provided.

Expansion joints should be treated in all structures intended to be tight with a joint filler sufficiently elastic to yield under the pressure exerted by the structure when expanding and keep the joint tight by flowing when it is contracting. Such a material is particularly advantageous in joints into which foreign substances may fall when opened and which might cause spalling of the concrete when it closes, as in roads, sidewalks, floors, etc.

Where water tightness is to be preserved, metal strips as described for construction joints should be used.

In floors of buildings, where trucking is to be done across the joints, sliding steel plates, set flush with the floor, covering the joint, are used to advantage to bridge the joint and also prevent fraying of the edges.

INSURING QUALITY CONCRETE IN COLD WEATHER

By F. H. McGraw[1]

Synopsis

The paper enumerates and describes the measures which may be taken to protect concrete against injury due to low temperatures in cold weather, and the conditions under which each of these measures should be applied.

To epitomize:

Concrete which has alternately frozen and thawed while setting will not harden.

Concrete does not harden while frozen.

Concrete sets and hardens more rapidly at temperatures above 50° F.

The minimum precaution permissible in cold weather is that which will safely protect the concrete against freezing.

Additional precautions to be taken and their extent depends upon the rate at which it is desired that the concrete attain its designed load-bearing strength.

To place concrete in cold weather, even to build large, important and expensive structures in the midst of the severe winters of our Northern climate, is no longer a novelty. It is done every winter in every large city and in many small ones and little is said about it. But to make concrete of the highest quality, equal to that produced under the more favorable conditions of summer weather, requires care and a high degree of control over conditions, which control may not with safety be omitted, neglected or relaxed.

It is the purpose of this short paper to outline the conditions which must be maintained to insure a quality product, rather than to deal with methods or apparatus or appliances used to secure and maintain these conditions. Before considering these essential conditions, it may be well to list a few well-established and generally accepted facts about the setting and hardening of concrete at low temperatures which have a direct bearing upon quality.

1. Concrete will develop its strength faster at higher temperatures and slower at lower temperatures.

2. If concrete is to attain its designed load-bearing strength it must be kept above the freezing point of the water used in gaging.

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[1] Construction Manager, Dwight P. Robinson and Co., New York City.

3. If its designed load-bearing strength is to be attained in a reasonable period of time the temperature must be many degrees above the freezing point; 50° F. is generally considered the point below which setting and hardening is greatly retarded.

4. Concrete does not develop strength to any considerable degree while frozen.

5. To permit concrete to freeze means practically to suspend its hardening process during the period that it remains frozen.

6. To permit newly placed concrete to alternately freeze and thaw during the setting and hardening period will seriously reduce its ultimate strength.

In the light of these facts, it is not difficult to determine rather definitely not only the nature but the extent of the safeguards and precautions which must surround concrete at low temperatures. The only safe procedure is to take measures which will positively insure against freezing, and if hardening at a normal rate is required to hasten construction progress or to strip forms in order to use them over again, the concrete should not be permitted to reach a temperature below 50° F.

The problem under consideration may, therefore, be divided into two parts: First, to prevent freezing of the concrete; and second, to maintain a temperature which will permit hardening to progress at an approximately normal rate. The first part of the problem may be accomplished by either of two methods or a combination of them: (*a*) to maintain the concrete above 32° F., and (*b*) to lower the freezing point of the gaging water by adding a chemical compound to it. The use of the former method or a combination of the two is usually the safest course to pursue. The use of chemicals to lower the freezing point of concrete is effective over a limited range. The chemicals most commonly used to lower the freezing point of the gaging water are sodium chloride (common salt) and calcium chloride. Salt lowers the freezing point but it also slows up the hardening process. Calcium chloride lowers the freezing point to a greater extent than salt when used in equal quantity, but it accelerates the setting and hardening of the cement. A reduction of the freezing point by about 10° F. is all that can be counted on by the use of calcium chloride without using such a quantity as would be injurious to the concrete. Should the temperature of the air drop below 22° F., freezing would result unless heat were applied.

In climates where the thermometer is likely to drop below 22° F., the safest course is to provide heat. Heat may be applied by two methods, either to the aggregates and water used in making the concrete or to the structure after placing. Whether both of these methods are to be used or only the former depends upon the minimum temperature to which the concrete is likely to be subjected during its setting and hardening period. When the weather is moderate and a sudden or large drop in temperature is improbable, it is often considered safe to rely upon heating the aggregates and water before mixing. They should be raised to a temperature of 100° F., but not above 150° F., so that the resulting concrete may have a temperature of 80° F. or more at the time of placing. Over-heating of some types of aggregate should be avoided since they are liable to be injured at high temperatures. A number of effective devices are available for the purpose of heating materials.

The chemical reactions which occur when cement sets and hardens produce heat. The result is a perceptible rise in the temperature of the concrete. When the mass is large this heating effect offers considerable assistance in preventing freezing. If the temperatures were only a few degrees below freezing and could be counted upon to go no lower, the preheating of the concrete materials and proper covering of the placed concrete to retain the heat initially in the concrete, plus the heat generated during the setting period, would probably be sufficient to protect the work. If in addition there is dissolved in the mixing water from 2 to 4 lb. of calcium chloride for each bag of cement used, the work might be considered reasonably protected against freezing at a temperature of 20° F. The phrase "reasonably protected" is used advisedly. In a high wind such concrete would freeze unless protected from it. But unfortunately our Northern climate is not so dependable and other precautions are usually necessary for safety. Inclosing the structure and applying heat to maintain a temperature surrounding the concrete that is always above the freezing point regardless of the outside temperature is the one safe protection against freezing in Northern climates in winter. If normal setting time of the concrete is the end sought then these same measures are used, but sufficient additional heat must be applied so that not only is the concrete kept above its freezing point but is maintained at a temperature above 50° F. for at least two weeks, and preferably longer, after placing.

Many types of inclosure that have been devised for retaining heat within and around concrete structures have been described and illustrated in articles in the technical press and in the literature of concrete construction. It is not within the scope of this paper to deal with them. Suffice it to say that since their primary purpose is to safeguard the quality of the concrete in the structure by maintaining favorable temperature conditions, it is of first importance that such inclosures should be designed and constructed with such thoroughness and care as will insure their performance of this function.

It is also important in such cases to insure against the too rapid drying of the concrete. The heat used to maintain the higher temperature not only quickens the setting but also accelerates drying, especially since the cold outside air is often of low relative humidity. When cold air, even of high humidity, is heated its relative humidity is greatly reduced. This warmed air, of low humidity, has a marked effect on the drying out of exposed concrete. Sprinkling should be practiced, in such cases, for a week or more after placing.

In conclusion, "quality" concrete, and permanent concrete, can be placed in cold weather if well-known and generally recognized precautions surround it and intelligent supervision and control are mixed into it. Quality concrete in winter or summer is not a mixture of cement, sand, stone and water, but requires a fifth component—intelligent supervision.

TRANSVERSE TESTS AS A CRITERION OF THE QUALITY OF CONCRETE

By H. S. Mattimore[1]

Synopsis

This paper aims to demonstrate the efficiency of the transverse test as a means of control for field concrete. Comparative results of transverse and compression tests are given in several curves and tables, one of which illustrates the comparative percentage variation of individual specimens in transverse and compression tests obtained in over fifty sets of specimens molded from field concrete.

As a result of data included in the paper, the following conclusions are drawn:

Since the modulus of rupture is used as a basis of design for all rigid pavements in highway work, the transverse test is largely replacing the compression test, and all indications are that in the near future it will be the standard method of control for concrete in the highway field.

Large series of comparative tests in cross-bending and compression demonstrate that more concordant results are obtained in the cross-bending tests than in compression tests on individual specimens made from the same mix.

The transverse tests have been found applicable to field control of concrete. Inexpensive field apparatus has been found to give concordant results. Therefore, from the standpoint of more reliable control and the economy involved, the transverse test should be given consideration for the control of all field concrete.

In judging the efficiency of a type of test to determine the quality of concrete for field control, the following are main factors for consideration:

1. The stress applied to the test specimens should have a definite relation to the stresses used as a basis for design of the structure;
2. The tests should give concordant results.

It has been definitely established[2] as a result of tests on experimental roads that the transverse stress is the critical stress in a road slab, and, hence, that the modulus of rupture of concrete is a most important property in determining the strength of the slab. The modulus of rupture is now used by highway engineers as the basis for design in all rigid pavements, and the determination of the flexural strength of plain concrete placed in road slabs has been given special consideration by highway engineers in the past several years. At first, attempts were made to establish a relation between compressive and flexural strengths, but no reliable constant relation could be established. A study of this problem, from many series of tests, indicated that the modulus of rupture varied from 16 to 25 per cent of the compressive strength, so that in order to be assured of concrete having high enough flexural strength, it is found necessary to subject it directly to a cross-bending test. This test is used not only for preliminary determination and control in the laboratory, but also for control in the field, suitable field testing apparatus having been developed.

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[1] Engineer of Materials, Pennsylvania State Highway Department, Harrisburg, Pa.

[2] See M. O. Withey, *Proceedings*, Sixth Annual Meeting of the Highway Research Board, p. 239 (1926).

As a result the transverse test is largely replacing the compression test in the highway field. The size of beam and the details for making this test are being investigated by committees of national highway associations, and when these are standardized there is very little question that the transverse test will be the standard adopted for the control of concrete in all highway work.

Where the flexural strength of concrete is a minor consideration in design, as in reinforced structures such as bridges or buildings, the choice between the compression test and the transverse test as a means of field control would depend upon the relative concordance of individual test results.

For many years, the compression test was used almost entirely both for original investigation in the design of concrete and for field control. Many investigators reported considerable variation in the test results of individual specimens made from the same mix. In a paper presented at the 1926 meeting of the American Concrete Institute, H. F. Clemmer and Fred Burggraf[1] give results of a series of tests consisting of thirty transverse and thirty compression tests which show an average variation in the transverse tests of 3.7 per cent, while the average difference in the compressive strength was 72 per cent. Other investigators report maximum variation in test results on individual compression test specimens molded from the same mix of 25 to 40 per cent.

After a study of many of these investigations, it seems reasonable to conclude that much of this difference occurs in the size and shape of the specimen, the method of making the specimens, and the method of testing. Referring to Table I, showing comparative results of transverse and compression tests made on the same mixes, it will be noted that the compression test shows no appreciable gain in strength as the fineness modulus and coarse aggregate content increases, but a considerable gain is noted in the transverse test results.

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[1] H. F. Clemmer and Fred Burggraff, "Transverse Testing of Concrete," *Proceedings*, Am. Concrete Inst., Vol. XXII, p. 304 (1926).

TABLE I.—EFFECT OF FINENESS MODULUS AND WATER-CEMENT RATIO ON TRANSVERSE AND COMPRESSION TESTS.

Data from *Bulletin No. 11*, Structural Materials Research Laboratory, Tables II and IV.
Each Value is the Average of Five Tests Made on Different Days.

Reference No.	Sieve Analysis: Amount Retained on Each Size, per cent by weight									Fineness Modulus	Water-Cement Ratio	Modulus of Rupture, lb. per sq. in.	Compressive Strength, lb. per sq. in.
	No. 100	No. 50	No. 30	No. 16	No. 8	No. 4	$\frac{3}{8}$ in.	$\frac{3}{4}$ in.	$1\frac{1}{2}$ in.				
30	99	88	57	46	41	35	25	9	0	4.00	0.98	390	2000
34	100	96	86	83	80	78	58	19	0	6.00	0.78	600	2250
35	100	97	89	86	85	84	63	21	0	6.25	0.77	590	1990
39	99	90	63	54	49	45	0	0	0	4.00	0.98	455	2110
40	99	93	75	69	64	61	39	0	0	5.00	0.87	560	2650

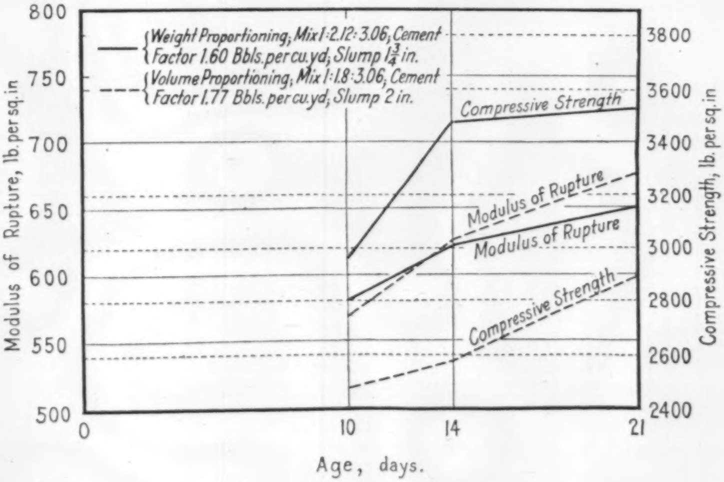

FIG. 1.—Comparison of Transverse and Compression Tests in Mixes with Different Sand Content.

Figure 1 is a comparison of transverse and compression test results made on the same aggregates in two different proportions, one by weight, the other by volume. In the latter, no allowance is made for the sand bulking, so that the sand content is about 20 per cent less than in the weight proportioning. The compressive strength of the concrete proportioned by weight, with high sand content, is considerably greater than that of the concrete proportioned by volume, with low sand content. The modulus of rupture, on the contrary, shows only a small difference favorable to the volumetric proportioning, or richer mortar with low sand content.

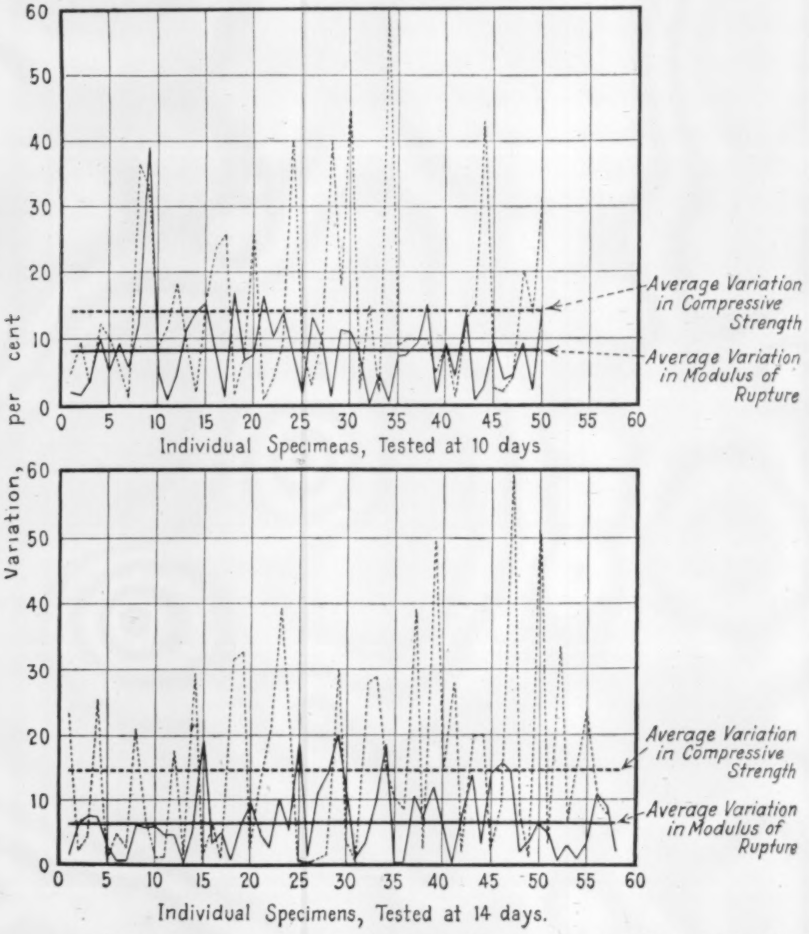

FIG. 2.—Comparative Percentage Variations in Transverse and Compression Tests of Concrete for Road Construction, Pennsylvania Department of Highways.

The comparisons in Table I and Fig. 1 show how the results of compression tests are influenced by the plasticity of the mix. Mixes with high sand content, due to the plastic character, give a more uniform specimen molded in a 6 by 12-in. cylindrical mold and up to a certain point, where the mortar is not too lean, have a tendency to give higher compressive strengths. As the coarse aggregate content increases it is more difficult to mold the specimen, and the compression test either shows loss or at least no appreciable gain, while at the same time the transverse test reflects the increase in strength due to larger stone content and richer mortars.

Both the transverse and compression tests as methods of control for field concrete were tried on many projects by the Pennsylvania State Highway Department during the construction season of 1926. Figure 2 is a comparison of the percentage variations of test results on transverse and compression specimens made from the same mix of concrete being laid in road slabs. The points plotted in each case are the variation obtained in tests on two individual beams in the transverse test and two individual cylinders in the compression test, made from the same batch of concrete, by the same operator. The average variation in the compression test at 10 days is 14.5 per cent and in the transverse test 8 per cent. However, the most striking feature in this comparison is the great difference in some of the individual compression test results. This difference in individual specimens is not realized when averages are reported in many series of published test results, but it may be vital in field control, where the usual practice is to make a limited number of test specimens.

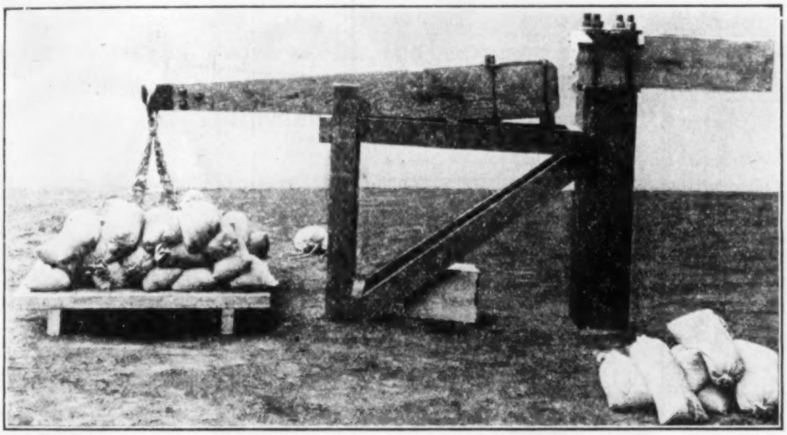

FIG. 3.—Field Testing Machine for Transverse Test of Concrete.

Figure 3 illustrates the field testing machine used for the transverse test by the Pennsylvania State Highway Department.

Figure 4 gives some of the detail assembly of the apparatus shown in Fig. 3. The test specimen is in the form of a rectangular beam 6 in. wide, 8 in. deep, and 40 in. long. This length is sufficient to obtain two tests on the same beam. The test specimen is clamped so as to allow an overhang of 1 ft., and the load is applied by means of an extension lever arm, resulting in a cantilever action.

The following formula for rectangular beams is used for calculating the modulus of rupture:

$$S_r = \frac{M}{\frac{bd^2}{6}}$$

where S_r = modulus of rupture;
M = maximum bending moment;
b = breadth of beam; and
d = depth of beam.

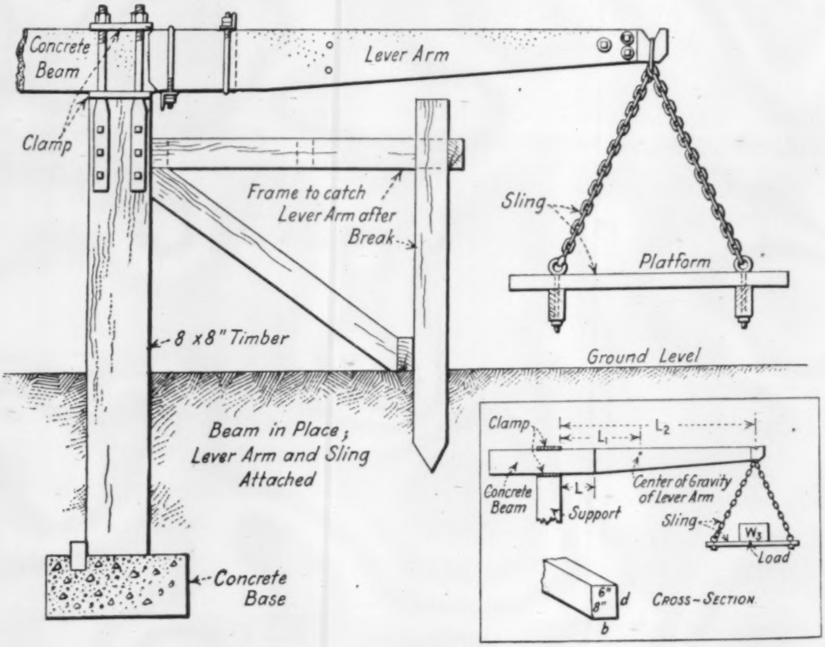
Concrete Beam
Clamp
Lever Arm
Frame to catch Lever Arm after Break
Sling
Platform
8 x 8" Timber
Ground Level
Beam in Place; Lever Arm and Sling Attached
Concrete Base
Clamp
L_2
L_1
Concrete Beam
L
Support
Center of Gravity of Lever Arm
Sling
W_3
Load
6"
8"
d
b
CROSS-SECTION

FIG. 4.—Showing Details of Transverse Testing Machine for Concrete.

Referring to Fig. 4, in calculating the value of M for substitution in the above formula, the following factors are used:

W = weight of the end of the beam broken off in test, approximately 50 lb.
W_1 = weight of lever arm, calibrated for each machine.
W_2 = weight of sling and platform, calibrated for each machine.

L = length of the end of the beam broken off, approximately 1 ft.

L_1 = distance from butt end to center of gravity of extension lever arm, calibrated for each machine.

L_2 = length of lever arm, calibrated (designed 64 in.).

W_3 = weight of load applied (variable).

Of the above factors, all can be considered as constants but W_3, the weight of the applied load. There is a slight variation in W and L in different tests, but it was found in comparing many results that this could be neglected in computation.

CONCLUSIONS

Since the modulus of rupture is used as a basis of design for all rigid pavements in highway work, the transverse test is largely replacing the compression test, and all indications are that in the near future it will be the standard method of control for concrete in the highway field.

Large series of comparative tests in cross-bending and compression demonstrate that more concordant results are obtained in the former than in the latter on individual specimens made from the same mix.

The transverse tests have been found applicable to field control of concrete. Inexpensive field apparatus has been found to give concordant results, so that, from the standpoint of more reliable control and the economy involved, this type of test should be given consideration for the control of all field concrete.

THE FIELD TESTING OF CONCRETE

By Roderick B. Young[1]

Synopsis

The conditions necessary for a test are:

1. A property that can be measured;
2. A standard of comparison;
3. A representative sample in a form that can be tested;
4. A method for conducting the tests, with the necessary equipment;
5. Results that can be interpreted.

Each of these is discussed briefly and it is shown that the present standard methods of the Society dealing with field testing of concrete are incomplete.

The interpretation of field results is considered from the standpoint of the factors that influence the test results. The accuracy of the design of the concrete can be definitely determined from the field tests, but the uniformity of the concrete is not so easily ascertained. Various methods used to evaluate the uniformity of concrete are reviewed and their interrelations and limitations mentioned. A factor based on the "progressive strength" curve is suggested as a measure of uniformity.

The relation between field concrete and field tests as found by different investigators is referred to, and the need of further investigations along this line brought out.

Testing is defined as "putting to the proof." The field testing of concrete, therefore, is putting concrete to the proof; concrete that is job-produced, proved by methods that can be applied under field conditions. Its purpose is to determine whether or not the concrete is of the desired quality, as measured by some accepted standard.

The conditions necessary for a test are:

1. A property that can be measured;
2. A standard of comparison;
3. A representative sample in a form that can be tested;
4. A method for conducting the tests, with the necessary equipment;
5. Results that can be interpreted.

The Measurable Properties of Concrete

Concrete has many measurable properties but not all meet the requirements of field testing, namely, that they be measurable by methods that are cheap, simple and readily available. The properties of concrete meeting these conditions are chiefly absorption, compressive strength, transverse or flexural strength, and consistency or workability. Each of these is in use at present. Absorption tests are largely confined to concrete products; the compression test to structural and highway work. The transverse test, while in limited use for years, has recently been adopted by a number of highway engineers and is becoming increasingly important in this field. Consistency or workability tests, as exemplified by the well-known "slump" test, are in general use for purposes of control while concreting is in progress.

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[1] Senior Assistant Laboratory Engineer, Hydro-Electric Power Commission of Ontario, Ontario, Canada.

The time element in construction influences the nature of the test that can be applied practically in the field; for modern conditions demand speed, and tests, to be useful, must give immediate results. Compressive or transverse strength is customarily determined when the concrete is 28 days old; but by this time the concrete represented by the test has become history, so that many organizations require tests at 7 days in an attempt to obtain information sooner. Even a 7-day test does not give data until the concrete represented by it is in the structure and its faults, if any, are past correction. The only test that gives information immediately is the slump test, which is used to measure the plastic properties of the mixture. The slump test at best can but indicate indirectly and very approximately the properties of the finished concrete, so that its value as a record of the quality of the concrete is strictly limited.

STANDARDS OF COMPARISON

While it is necessary to refer to the part standards play in field testing, yet their detailed consideration is outside the scope of this paper. Standards of comparison for field testing should be stated in terms of properties that can be measured in the field, but the degree to which these properties must be present in any given case is determined beforehand by the engineer responsible for the design of the structure in question. Standards for the properties of concrete should not be confused with the standard methods for testing, of which more will be said later.

THE SAMPLE

No test is better than the sample upon which it is made. The sample should be truly representative of the material of which it purports to be a part. The Society has promulgated a standard method of taking and preparing samples of field concrete[1] which in brief provides for:

(*a*) A sample taken from the work "immediately after the concrete has been placed;"

(*b*) The molding of a sample into a cylindrical specimen of length twice its diameter;

(*c*) The molding of the specimen by a puddling process;

(*d*) Curing "in damp sand until the date of test."

The Standard Method has received general adoption in most particulars. It is an excellent guide but incomplete and it does not fully cover present-day practice. For instance, it is doubtful if the method of sampling as specified there is being generally followed. Few engineers sample the concrete after it has been deposited. The author is not familiar with conditions on highway work, but in the structural field he has observed that sampling is more often done at the mixer, at the hopper or from the buggies than from the forms.

Sampling from the forms has the advantage that the sample is representative, not of a single batch but of a mixture of batches as it exists in the structure. This is true in mass placing, but as the sections become of less size, the form-taken sample becomes more and more a sample of a single batch, while in road work the form-taken sample is always a batch sample. There are locations, such as in heavily reinforced sections, where it is difficult, if not impracticable, to sample from the form and other cases, as just cited, where the two methods of sampling are equivalent. The author favors sampling from the forms, believing it to be sounder practice, but there is good precedent for the other method and many prominent concrete engineers regularly permit batch sampling on their work.

The temperature of curing is another point not fully covered by the Standard Methods of the Society. Most engineers dealing with concrete are familiar with the effect of temperature on curing. Few realize, however, how extremely sensitive to temperature is a small mass of concrete, such as a 6 by 12-in. test cylinder. Experience has shown that unless concrete test specimens are kept at approximately the same temperature as used in laboratory testing, the results will not be reliable. In the field, conditions are such as to make the maintenance of a proper storage temperature a matter of the greatest difficulty, particularly in cool or cold weather; yet unless a temperature averaging 60 to 70° F. is maintained, the tests are almost valueless as an indication of the quality of the concrete they represent.

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[1] Standard Method of Making and Storing Specimens of Concrete in the Field (Serial Designation: C 31 – 27), 1927 Book of A.S.T.M. Standards, Part II.

Another point not fully treated in the Standard Methods of the Society is the preparation of the ends of the specimen. A method of capping is given, but the importance of a proper cap is not indicated. The condition of the ends of the specimens has a very important influence on the results obtained. Gonnerman[1] has found that a convexity of 0.01 in. reduces the compressive strength of concrete test cylinders from 20 to 35 per cent. He studied the effect of other abnormal end conditions and came to the conclusion, "That too great care cannot be exercised when preparing cylinders for test in order to secure ends which are true planes."

The author's experience has been that it is not difficult to get field specimens of concrete properly molded, nor to provide damp storage. It is more difficult to enforce sampling from the forms and to get field inspectors to appreciate the necessity for absolutely plane ends on the specimens, but the matter of greatest difficulty, particularly in work situated at some distance from a laboratory, is to maintain proper storage temperatures and this can only be had at the expense of considerable time and forethought and by constant supervision.

There are two distinctly divergent view points as to how concrete samples should be cured. There are those who believe that test specimens should be cured under job conditions, receiving the same treatment that is received by the concrete they represent. This view is predicated on the idea that, if the sample receives the same treatment as the structure, its strength will be identical with similar concrete therein. Tests made in this way are useful to indicate when it is safe to strip forms, remove shores, open a road to traffic or for some similar purpose. The weakness of this system of testing lies in the fact that a given set of curing conditions will affect the small specimens differently than the larger bodies of concrete it represents. The method, too, has a serious fault; the results obtained are partly related to the inherent quality of the concrete and partly to the subsequent treatment the specimen receives. In a given case it is always difficult to determine the degree to which each factor operates.

The other view point held is that the specimens should receive a standard treatment, such as is provided in the Standard Methods of the Society. The theory underlying this attitude is that specimens cured under carefully controlled conditions are an index of the inherent quality of the concrete—the optimum quality, that with proper treatment, the concrete will possess. The advantage in this system of curing is that the tests are comparable with similar tests elsewhere or to laboratory tests made by similar methods. It agrees with the established principles of testing, that the tests on a material should disclose the properties of that material and that supplementary tests or subsequent inspection should be depended upon to see that the material is not abused while it is being fabricated.

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[1] H. F. Gonnerman, "Effect of End Condition of Cylinder in Compression Tests of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 1036 (1924).

METHODS OF TESTING

The standards of the Society do not provide for the actual testing of field concrete, and presumably laboratory standards are to be followed. Laboratory standards require that concrete be tested damp as removed from storage. No such provision exists for tests made on field specimens and the author has found it a not uncommon practice to dry out field samples one or more days prior to crushing. The results obtained by the latter treatment are from 10 to 20 per cent higher than would be obtained by testing damp and are not comparable with those obtained by following the standards in force in the laboratory.

Laboratory standards do not permit cushioning the ends of the specimens when testing, but Gonnerman[1] has shown that under certain conditions Beaver board can properly be used for this purpose and Purrington and McCormick[2] have shown that bedding with a sand cushion is satisfactory. Whether or not these practices should be forbidden is a debatable point that might well be considered by the Society, since at times some form of bedding can hardly be avoided. The practice of cushioning is almost universal and will continue so until definitely prohibited by an authoritative specification.

Usually, field specimens of concrete are sent to some established laboratory for test, in which case standard testing equipment is used. There are jobs, however, outside the big centers where suitable facilities are not readily available and some other solution must be found in these cases. This is particularly necessary where the work is so situated that the conditions of transportation are uncertain and out of the control of the engineers, as would be the case where long shipments have to be made by express. The Hydro-Electric Power Commission of Ontario has met this difficulty by installing a field laboratory wherever the size of the project warrants. This laboratory is equipped for testing concrete in compression and for the various tests of fine and coarse aggregate, excepting that for mortar strength. The principal equipment is an hydraulic press of 100-ton capacity and the necessary sieves, scales, molds, etc. The cost of this equipment is less than 700 dollars of which about 500 dollars is for the press.

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[1] *loc. cit.*

[2] W. F. Purrington and James McCormick, "A Simple Device to Obviate Capping of Concrete Specimens," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 488 (1926).

INTERPRETATION OF THE TEST RESULTS

The interpretation of test results is a matter of some difficulty requiring judgment, a knowledge of testing with an appreciation of its sources of error and some experience with the methods of concreting. The results may be higher or lower than expected, consistently uniform or widely variable, and each condition has its significance.

Low test results may be due to the concrete, the specimen or the methods followed in testing, and the engineer must determine which is to blame. Examination of the specimen, where possible, will show the condition of the ends and any gross segregation in the cylinder. Faulty sampling is a possible source of unsatisfactory test results, although it can seldom be proved in any given case. Low tests from these causes can only be eliminated by exercising more care in sampling and making the specimen a matter of supervision and education.

Faulty curing, and ordinarily this is curing at too low a temperature, can be detected if the storage is equipped with a thermometer; its correction is obviously a matter of providing means to maintain the storage at the required temperature. Low temperatures are not a summer problem, but in the more northern latitudes become a factor early in the fall, when the nights become cool, and continue until the late spring. Another curing condition to be guarded against is the drying out of the specimen prior to placing in damp storage, during transportation or while waiting for test.

If the concrete is at fault, the most likely source of trouble is the use of excess water or of an improper water-cement ratio. Less frequent causes are poor cement or aggregates, the use of harsh mixtures, either due to a lack of fine aggregate in the mixture or, more rarely, to too dry a consistency. The field tests cannot, unaided, tell the engineer which of these is the cause and he must determine this from other data and from his personal knowledge of the job and the materials used.

Daily reports of the concrete work can be of considerable assistance in the interpretation of field test results, especially if they are purposely arranged to give information on materials and job conditions, Two examples of where this was true will be cited in illustration. In each case, field-control methods were in force and the concretes were designed closely for the strengths specified.

Case A.—The tests regularly fell below the design strength, sometimes only a few per cent but more often seriously. Conditions of sampling, curing and testing, and the use of excess water were eliminated in turn as causes, and it was discovered that the fault lay in the aggregate. The aggregate was a washed pit-run gravel containing a coarsely graded sand. Whenever the material lacked sand or the sand became very coarse, the strength fell away and could not be regained by the addition of any reasonable amount of cement. A study of the field data gave the first clue to the cause and a subsequent laboratory investigation confirmed the condition suspected as the probable reason.

Case B.—Sub-standard tests would occur in groups. The job was equipped with a weight-proportioning plant and was most carefully supervised. Job conditions were above the average. As in the previous case, the usual causes of low tests were eliminated one by one and the blame finally laid to one of the two brands of cement used.

The relation between the strengths obtained and those used in designing is a measure of the accuracy of the design. A mixture is not being closely designed if, when concrete testing 2000 lb. per sq. in. at 28 days is wanted, concrete testing 3000 lb. per sq. in. is regularly obtained. It is, of course, a safe result and may be satisfactory to the engineer; but it is not an accurately designed concrete, neither is it an economically designed concrete. On the other hand, if concrete is wanted, the strength of which is always to exceed 2000 lb. per sq. in., then it is proper to design for a higher strength, say 2200 or even 2500 lb. per sq. in., depending on job conditions, in order to provide a margin of strength sufficient to guarantee this result. However, this is not concrete designed for 2000 lb. per sq. in., but concrete designed for the higher strength and should be so considered.

The test data offers immediate information upon the accuracy of the design of the concrete mixtures, but is of limited use in determining that equally important factor, their uniformity. There is, at present, no standard method for rating the uniformity of concrete by means of its test results. Different schemes have been used at different times, such as the mean variation of the tests from the average strength, the percentage of tests that fall within a certain range of strength, or the plotting of the data in the form of "progressive strength" curves.

It might be helpful here to consider the factors that influence the uniformity of concrete. They are of several kinds: those that operate to vary the concrete from batch to batch, such as the measurement of materials and the moisture content of the aggregate; those that operate from hour to hour or day to day, such as changes in quality of the cement or aggregates; and those of less frequent occurrence, such as changes in the sources of the materials or in the methods or plant used in concreting.

The engineer responsible for field concrete endeavors to obtain a material that is uniform in quality and properly designed. He is not greatly concerned if the concrete to-day averages a hundred pounds per square inch greater or less than that placed a day or a week ago, provided both meet the specification. He considers uniformity in terms of changes from batch to batch or from hour to hour and field tests offer him next to no information upon variations within these short ranges of time.

Field testing as ordinarily carried out can be influenced but little by the batch-to-batch or hour-to-hour variations and this is particularly true where the specimens are taken from the concrete in the forms. It follows, therefore, that any figure derived from a series of field tests to express their degree of variability can measure only one type of uniformity, that is, the general uniformity of the concrete over the period of the job, but cannot indicate the batch-to-batch uniformity unless indirectly due to the fact that the same forethought that provides the first will usually provide the second.

A series of tests reflects not only the uniformity of the concrete but the care used in sampling, molding, curing and crushing the specimens. In the most carefully conducted laboratory tests there is a considerable variation in test results for specimens as similar in all respects as it is possible to make them. In a study of data from several well-known laboratories it was found that tests made by them, following the standard methods of this Society, gave mean variations in a set of similar cylinders ranging up to 25 per cent with an occasional value even higher, and that for large groups of data involving a number of variables, the average mean variation was from 7 to 11 per cent. The mean variation where the test specimens were made at one time averages slightly lower, 6 to 8 per cent.

Field tests appear to have mean variations of somewhat less magnitude: why is not just clear. Slater and Walker[1] report for the 28-day field tests made by them at Camden and Newark Bay, an average mean variation for sets of three cylinders, ranging from 0.4 to 25 per cent and averaging 2.9 and 3.1 per cent at Camden and 5.2, 5.9 and 6.4 per cent at Newark Bay. Corresponding data obtained by the Hydro-Electric Power Commission of Ontario have averaged about 5 per cent for groups of two specimens.

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[1] W. A. Slater and Stanton Walker, "Report of Field Tests of Concrete Used on Construction Work," *Proceedings*, Am. Soc. Civil Engrs., January, 1925, p. 3.

In field tests of concrete one has to consider not only the variations in the individual groups of tests but also that of the tests as a whole. The tests have been made from day to day,—singly or in groups of twos and threes,—and irrespective of any differences in the concrete are subject to a further disturbing factor, that of differences in curing. Laboratory tests made under these conditions have mean variations averaging from 7 to 11 per cent and it is reasonable to expect that the variations for field tests under similar conditions would be equally as great, that is, the probable variation for tests where the concrete was the same would be of like amount. If besides, the concrete is variable, then the mean variation of the tests made therefrom can be expected to be of even greater magnitude.

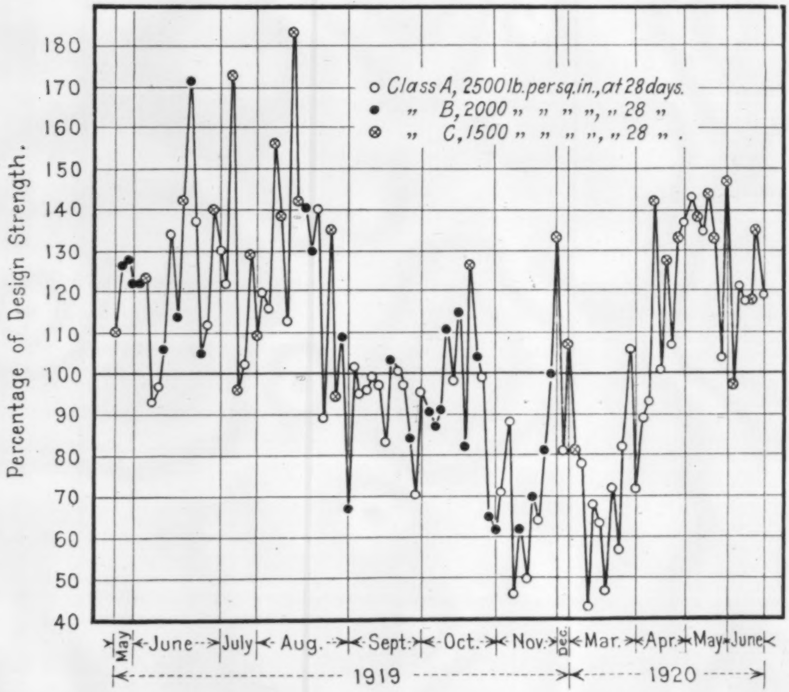

FIG. 1.—Field Tests, Nipigon Development, Hydro-Electric Power Commission of Ontario.

The mean variation of a series of tests from its grand average will be affected by the number of specimens taken. A job on which but a few tests are made is liable to show a high mean variation if one or two groups are abnormally high or low, whereas, if more tests were made the effect of these exceptional results would be relatively less. Of course, there is the possibility that the frequency of these abnormal results bears some relation to the number of tests taken, in which case the influence they exert on the mean variation of the series would always be the same. This is probably true where the number of tests runs into the dozens, but where, as is often the case, only a few groups of tests are taken, the influence of one or more abnormal results becomes quite marked.

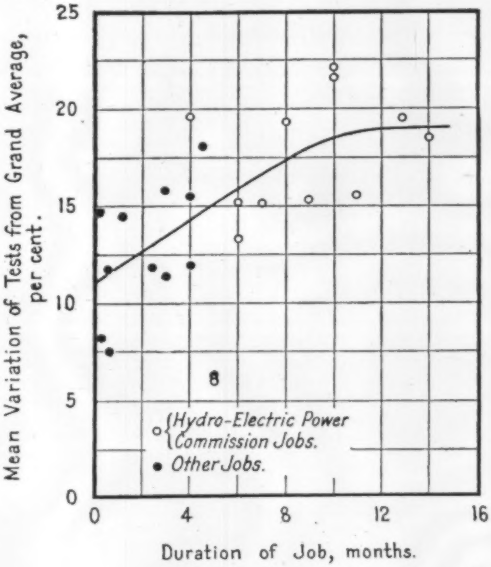

FIG. 2.—Influence of Time on the Mean Variation of Tests from Their Grand Average.

The duration of the job is an even more important factor. In a job covering a year, the average strength of the concrete for the first six months may be lower or higher than for the second, although both may meet the specifications. This may be due to differences in cement or aggregates, to different curing conditions or to some other factor. The variations for either of these periods would be less than for the two periods combined and if the period of time was reduced still more, to say a few weeks, the average variation should be lower still. That time can be a factor can be illustrated from the data of Fig. 1. The tests there group themselves naturally into four periods,

TABLE I.—DATA FROM FIELD TESTS OF CONCRETE.

Name of Work	Location	Period Over Which Tests were Taken	Number of Specimens in 28-Day Tests	Compressive Strength, per cent of design strength	Mean Variation from Average of Like Tests in Single Set of Cylinders	Mean Variation of Average of Individual Sets of Tests from Average of All Tests	Percentage of Tests Falling Between 80 and 120 per cent of Average Compressive Strength	Constants from Progressive Strength Curves			
								Spread, per cent	Range, per cent	Slope, per cent	Uniformity Factor, Range ÷ Slope
PART I.—JOBS OF HYDRO-ELECTRIC POWER COMMISSION OF ONTARIO											
High Falls Development	Dalhousie, Ont	10 months	168	104	...	22.1	60	138	37	35	1.06
Nipigon Development:	Hydro, Ont.										
Power House, 1920		14 "	214	106	4.9	18.5	67	123	41	38	1.08
Main Dam, 1921		4 "	118	100	5.2	19.7	58	96	83	67	1.24
Queenston-Chippewa Development:	Niagara Falls, Ont.										
Screen and Power House		24 "	334	115	4.8	12.0	83	102	88	45	1.95
Canal and Forebay		13 "	636	117	...	19.5	66	144	56	57	0.98
Intake		5 "	38	116	4.0	6.0	100	42	90	23	3.91
Ranney Falls Development	Campbellford, Ont	10 "	212	81	5.9	21.7	54	149	80	72	1.11
Dam 8 Development	"	8 "	64	96	6.8	19.3	62	103	89	56	1.59
Dam 9 Development	"	6 "	62	95	8.6	13.3	81	105	68	31	2.20
Extension to Queenston Power House	Queenston, Ont	27 "	172	108	...	6.2	97	53	82	17	4.81
Extension to Nipigon Power House:	Hydro, Ont.										
Units 3 and 4		9 "	58	104	4.7	15.3	76	65	90	57	1.58
Units 5 and 6		11 "	164	98	4.9	15.5	75	120	95	61	1.56
Hanna's Chute Development	Bracebridge, Ont	6 "	30	142	4.6	15.2	67	64	40	21	1.91
Virgin Falls Storage Dam	Orient Bay, Ont	7 "	68	115	5.4	15.1	85	85	76	31	2.46
PART II.—JOBS REPORTED FROM VARIOUS SOURCES											
Building No. 10, Victor Talking Machine Co	Camden, N. J	11 weeks	267	116	3.0	15.8	79	139	73	48	1.52
Piers, Newark Bay Bridge, Central R. R. of N. J	Newark, N. J	10 "	264	108	5.8	11.8	78	92	82	41	2.00
Reinforced Concrete Building, New York Telephone Co	Brooklyn, N. Y	5 "	76	129	4.8	14.4	72	73	82	50	1.64
Addition to Stadium, Polo Grounds	New York City	1 "	26	117	2.1	8.2	100	34	100	35	2.86
Addition to building, Ward Bakery Co	New York City	2 "	46	109	3.2	11.7	83	50	70	34	2.06
Warehouse R. H. Macey Co	Long Island City, N. Y.	1 "	16	103	1.8	14.7	62	61	62	31	2.00
Foundation of building, New York Telephone Co	New York City	2 "	15	88	...	7.5	94	40	67	17	3.94
Office Building, Canada Cement Co., Ltd	Montreal, Que	5 months	84	102	3.0	6.1	100	30	64	14	4.56
Ross-Ode Stadium, Purdue University	Lafayette, Ind	3 "	81	...	...	11.3	85	59	52	26	2.00
Four concrete bridges, Bronx Parkway Commission	New York City	....	32	114	...	15.7	88	56	72	38	1.95
Seven bridges, California State Highway Commission	California	....	58	...	...	10.3	91	45	81	37	2.19
Stadium. University of Illinois	Urbana, Ill	4 months	177	...	...	11.9	75	96	85	44	1.93
Pavement, New Jersey State Highway Commission	New Jersey	4 "	32	...	...	15.5	62	104	66	47	1.41
Office Building, Portland Cement Association	Chicago, Ill	4½ "	165	147	...	18.1	64	135	81	64	1.27
Pittsburgh Stadium	Pittsburgh, Pa	....	485	106	...	12.9	85	80	84	44	1.91

the summer period ending August, 1919, the fall period ending October, 1919, the winter period ending early in April, 1920, and another mild weather period finishing the job. The mean variation for these periods are 17.4, 10.2, 15.9, and 14.9 per cent, respectively; that for the whole job 18.5 per cent.

Further confirmation of the effect of time is contained in Fig. 2 plotted from the data of Table I. There is a fairly definite relationship between time and mean variation for these jobs. There is a break in the relationship occurring at about one year, as seems reasonable since, after a certain length of time, the variables influenced by time would probably recur with the same frequency and magnitude as in the previous year and would thus have little additional effect on the mean variation.

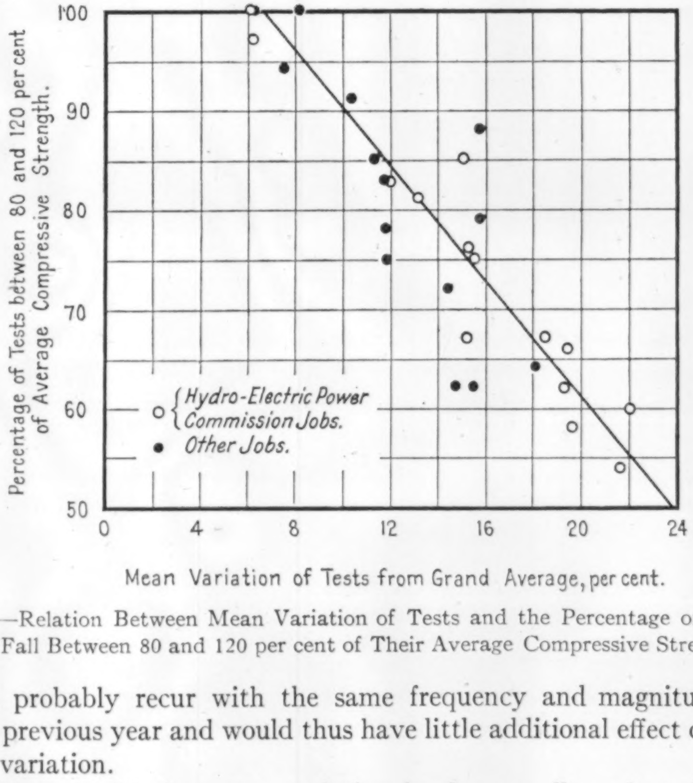

Fig. 3.—Relation Between Mean Variation of Tests and the Percentage of Tests that Fall Between 80 and 120 per cent of Their Average Compressive Strength.

What should the mean variation be for a well-conducted job? This is a difficult matter on which to express an opinion. The author believes that on work extending over a considerable period, say six months or more, and so situated that field laboratories must be used, concrete of more than average uniformity will have a mean variation of from 12 to 15 per cent, and that under more favorable conditions such as where established laboratories are available, this variation for equal concrete would be from 10 to 12 per cent. On jobs covering

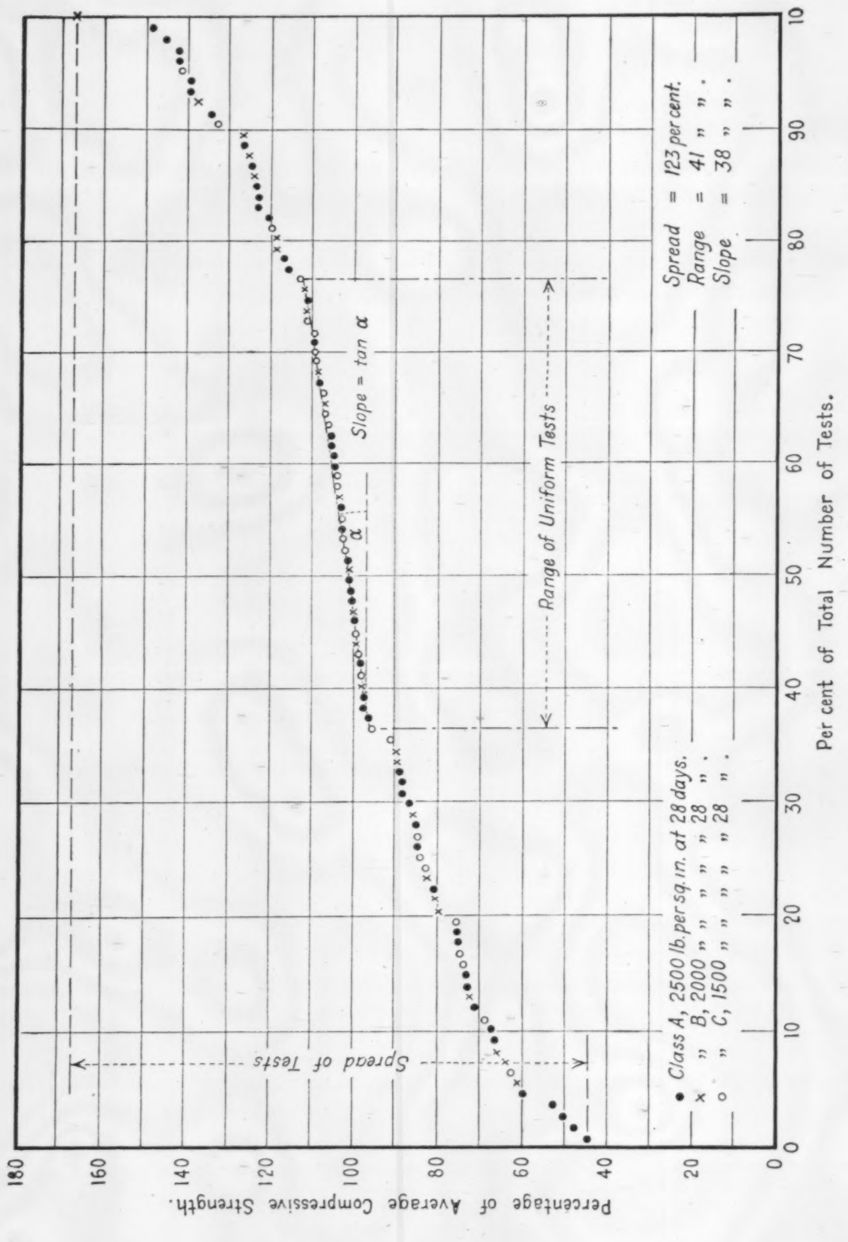

FIG. 4.—Progressive Strength Curve for Field Tests of Nipigon Development, Hydro-Electric Power Commission of Ontario.

periods of but a few weeks, the average variation should be less, say from 8 to 10 per cent.

Table I is offered in support of this opinion. Part I gives results obtained by the Hydro-Electric Power Commission of Ontario over a period of eight years on jobs on which field control of concrete was applied. In Part II are results assembled from published information from other sources. In connection with Part II it should be noted that with the exception of the California bridges, every job was handy to a regular laboratory, and on the first seven jobs, the tests were made as an experiment to determine whether or not field testing and control were practicable. The testing on the latter was done by laboratory experts assigned to the jobs for this purpose only, and on several the tests covered but a part of the work on which they were taken. Under these favorable conditions, the resulting mean variations were as high as 18 per cent.

The method of expressing the uniformity of test data in terms of the percentage of tests that fall within a certain range of strength is subject to the same limitations as those discussed as applying to the use of the mean variation. Generally speaking, the two are related in a rather definite manner as is shown by Fig. 3. Most of those who have reported results of test data have favored this method of stating uniformity and it probably gives a better idea of the variability than does the mean variation. With a mean variation of 11 per cent, it might be possible to have half the tests with no variation and half with 22 per cent variation, giving 50 per cent of the tests outside the 20-per-cent range; but from the standpoint of both the designing and field engineer responsible for the performance under the specification, there would be a great difference in a job where about 85 per cent of tests fell between 80 and 120 per cent of the average strength and where only 50 per cent came within the same range.

Bloecher,[1] Slater and Walker[2] and others have used so-called "progressive strength" curves to indicate the variability of field data. An example of such a curve is given in Fig. 4, which has been developed from the same data used for Fig. 1. This type of curve may be used to indicate:

(*a*) The extreme range of the variations;

(*b*) The normal range of the variations, either from the slope of the straight-line portion of the curve or from the intersection of the projections of this line on the vertical limits of the figure; and

(*c*) The relation of the more normal tests to those whose variation from the average is extreme.

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[1] W. P. Bloecher, *Proceedings*, Am. Concrete Inst., Vol. XIX, p. 100 (1923).

[2] *loc. cit.*

In this type of curve both the slope of the straight line section and the proportion of the data falling on the straight line are related to the uniformity of the concrete, but each has a different significance. If the concrete were absolutely uniform and the tests did not vary, the curve would be a straight line parallel to the base. As the concrete varies the line departs from the horizontal and the greater the variation of the concrete, the steeper the slope. A certain "slope" denotes a certain normal variation or range for that job and has much the same significance as the average variation already discussed. The two are in fact roughly related but the "slope" is not apparently affected as directly by such factors as the duration of the job, freak tests or periods of abnormal variation.

The curved sections at each end of the straight line may be caused by freak tests but apparently are more often due to abnormal variations in the concrete. The ratio of the straight-line section to the total curve, or the "range" as it has been called in Table I, indicates something of the success of the organization in maintaining uniform conditions of manufacture for the concrete. It does not measure the average uniformity of the concrete,—that is indicated by the slope of the curve,—but it does show approximately what proportion of the time this uniformity was obtained. On a well-organized job with skilled supervision, this ratio should be high, irrespective of what the slope happens to be.

The overall uniformity of the concrete is the combined result of the average uniformity and the time this average uniformity functioned. To denote this the author proposes a factor obtained by dividing the "range" by the "slope," or if u is taken as the uniformity factor,

$$u = \frac{R}{S}$$

where R is the percentage of the tests falling on the straight-line section of the progressive-strength curve, and S is the tangent of the angle that the straight-line section of the progressive-strength curve makes with the horizontal.

Where possible, values of u have been calculated for each of the jobs of Table I. As far as the Hydro-Electric jobs are concerned, and these are the only ones of which the author has personal knowledge, the uniformity factor varies with the known facts as to the relative uniformity of the concrete on the different jobs. This is truer of the uniformity factor than of either the "range," "slope," mean variation or the percentage of tests between 80 and 120 per cent of the average strength, and for this reason the author suggests this factor for further consideration as having possibilities as an index of uniformity of field concrete.

RELATION OF FIELD TESTS TO THE STRENGTH OF THE CONCRETE

The question is often asked, "What relation exists between the strengths obtained by testing field cylinders and that of the concrete in the structure?" This is a pertinent question, for the whole practice of field testing is based on the assumption that the tests there made are a measure of the quality or strength of similar concrete in place.

Such an assumption seems reasonable but test data bearing on the question are meager. In tests made in 1915 by the New York Public Service Commission,[1] concrete prisms cut from a 12-in. wall gave compressive strengths some 25 per cent greater than similar specimens made and stored in the laboratory. Mattimore[2] published data on a large number of cores taken from road slabs. He found a general tendency towards considerably higher strengths in the drilled specimens than in the molded specimens. He stated as his opinion that "the strength of the molded specimen is not always a positively reliable indicator of the strength of the concrete in place as determined by the drilled specimen." Gage[3] at a later time, went even further in condemning the field molded specimen, for he was of the opinion that "there does not appear to be any definite relation between the strength secured on samples prepared during construction and the strength shown by the cores cut from the same pavement after construction."

On the other hand, a contrary finding has been made in two cases on record where a careful comparison between field molded and drilled specimens of similar concrete has been made. The first of these is the investigations made by Slater and Walker[4] for the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete. These investigators concluded as a result of their tests that "in general, the strength at 28 days and 3 months of molded specimens cured in damp sand, were about equal to or slightly greater than the strengths of specimens cut from test slabs and columns which had stood in the building with no treatment other than that to which the building itself was subjected." In the second of these investigations reported by Bragg[1] a direct comparison was made between molded and drilled specimens. The average of 32 tests made at different times was practically the same whether the specimens were molded and field cured, molded and laboratory cured or drilled. While Bragg offers no such opinion, the logical conclusion from his tests is that the concrete in the specimen closely represents the concrete in the structure, under the conditions of curing existing in pavement work.

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[1] *Engineering Record*, September 4, 1915, p. 296.

[2] H. S. Mattimore, "Relation Between Molded and Core Concrete Specimens," *Engineering News-Record*, January 12, 1922, p. 73.

[3] R. B. Gage, "Conclusions from Wm. Dentt Tests of Concrete Road," *Engineering News-Record*, August 9, 1923, p. 226.

[4] *loc. cit.*

CONCLUSIONS

Enough has been said to show that not all is known about the field testing of concrete. There is need both for further research and for further standardization in this field. It is one particularly within the province of this Society and the writer believes the time has come when the Society through the proper committee might with profit do both.

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[1] J. G. Bragg, "Comparative Utility of Concrete Pavement Tests," *Engineering News-Record*, June 12, 1924, p. 1012.

DISCUSSION ON FIELD CONTROL OF THE QUALITY OF CONCRETE

Discussion on Proportioning

Mr. Conner. MR. C. N. CONNER.[1]—I should like to ask if Mr. Crum has made any comparative strength tests between proportioning fine aggregate for concrete by inundation and by weight; a comparison of strength tests by using those two methods.

Mr. Crum. MR. R. W. CRUM.[2]—I do not know of any data on that. From the theoretical standpoint, if the operation is correctly done in each case, the strength should be the same if the constituents and the mixture of the two concretes are the same.

Mr. McMillan. MR. F. R. MCMILLAN.[3]—We have made some tests in our laboratory in which the fine aggregate was proportioned both by inundation and by dry volumetric measurements. When corrected for the water absorbed, the water-cement ratio strength law applied equally well to the two sets of data.

Mr. Gilkey. MR. H. J. GILKEY[4] (*presented in written form*).—Mr. Crum's treatment should go far toward clarifying for many the two leading present-day theories of design. That most widely used, the water-cement ratio theory, represents a great forward step in the use of concrete. It seems very probable, however, that this so-called law does not fully include all that enters into the matter of concrete design. The zone is unduly wide and it seems probable that there are many other laws within this law. To assert, as there seems to be a tendency to do in some quarters, that any such empirical relation, or so-called law, embodies the alpha and omega of concrete design savors of a dogmatism that it is difficult to accept in the light of the great range of strengths attainable under the law. Would it not be better for us to be a little less positive and a little less inclined to burn our bridges behind us? Make the maximum use of the information at hand but maintain an open mind for further refinements. Some of these are worthy of mention here.

To what extent are variations in the results from the law dependent upon the kind, amount, and grading of aggregates; the particular brand of cement being used; differences in manipulation; leakage from molds or forms after placement; and temperature effects in

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[1] National Research Council, Washington, D. C.

[2] Engineer of Materials and Tests, Iowa State Highway Commission, Ames, Ia.

[3] Director of Research, Portland Cement Association, Chicago, Ill.

[4] Associate Professor of Civil Engineering, University of Colorado, Boulder, Colo.

Mr. Gilkey. curing? Can fair comparisons be made between mixtures of equal water-cement ratios but widely different workability? Can fair comparisons be made between mixtures of equal water-cement ratios and equal workability but different aggregate-cement ratios?

The technique of applying to field conditions the laboratory knowledge that we possess is indeed the greatest present need in concrete. The Portland Cement Association has been performing a great work in the educational program that it has conducted along these lines. Second only to them are such valuable endeavors as the present symposium. Much remains to be accomplished but encouraging progress is being made at a rapid rate.

Early Strength Concrete.—As Mr. Crum implies, early-strength concrete is merely high-strength concrete. The knowledge most needed in this connection is information as to what strength may safely be expected at the age of one, three, or seven days for different cements and mixtures. The cooperative tests under the auspices of Committee C-1 on Cement should furnish some information of value as regards variation in the rate of strength gain in different cements. It is only a beginning however.

Mr. Ahlers. MR. JOHN AHLERS[1] (*presented in written form*).—The following discussion is entirely from the angle of the contractor attempting to solve in an engineering manner the problem of field control of concrete. For the purpose of simplicity of field procedure, the water-cement ratio has been found the most simple to control and the easiest by which to design concrete mixtures for definite and predetermined strength. As stated by Mr. Crum, both the water-cement ratio and the void-cement ratio fundamentally necessitate a careful proportioning between the water and the cement and this is the feature on which our contracting organization has concentrated its efforts.

Our problem has been solved by the simple arrangement of a mechanical device weighing each batch of cement against its proportionate and proper amount of water, eliminating the variables due to uncertainty in measurement of water, and uncertainty in amount of cement in bags, shaking of bags and broken bags. The device has the added advantage of simplicity provided for increasing and decreasing workability without changing the strength.

Mr. Crum rightly states that improvements are being made in water-measuring devices, and also points out the necessity of accurate knowledge of the moisture in both the fine and coarse aggregate.

By taking an amount of water 0.5 per cent in excess of that found existing in sand and gravel, sufficient margin exists for any possible variation within one-half to one hour's time on the job. **Mr. Ahlers.** Larger variations are easily discerned and can be verified by test.

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[1] Barney-Ahlers Construction Co., New York City.

Mr. Crum rightly points out that different conditions affect the strength of concrete existing on every job. Such factors as the quality of the cement, the quality and cleanliness of sand, quality and cleanliness of aggregate, time of mixing, etc., all affect the strength of the concrete and the water-cement ratio curve.

The procedure followed by our contracting organization has therefore resolved itself into establishing so-called "job curves" for every operation. When one or two points are found on this curve it will always be parallel to the water-ratio curve and the desired water-cement ratio for the strength wanted in the structure can be arrived at graphically and mechanically without computations by the use of a diagram which is the basic principle of a mechanical device used throughout our concreting operations.

DISCUSSION ON CONVEYING AND PLACING

Mr. Walter. MR. L. W. WALTER.[1]—Referring to the paper by Mr. Doe, and more particularly to that part coming under the head of Effect of Admixtures on Conveying and Placing Concrete, the author states that "Hydrated lime has been added to concrete on various classes of work for years and no detrimental effects have been found. Celite is a comparatively new material, and its effects on such qualities of concrete as strength, absorption of moisture, weathering, etc., are still under observation." Following that, examples are given of the various classes of concrete in which these materials may be used advantageously, and a little further on reference is made to other classes of work in which, by the omission of celite, inference might be drawn that celite is not so well suited as is lime in concrete for certain particular requirements, such as exterior walls exposed to severe weather conditions, concrete designed to resist water pressure, or concrete requiring special strength.

Unquestionably, concrete of which ships were constructed was designed to meet such exposure as would commonly be met with in exterior wall construction, was designed to resist water pressure, and required special strength. In the examination made in 1926 of the concrete in a ship built and launched in 1918, a structure in which, by design, reinforcing steel was placed within $\frac{3}{4}$ in. of the outside surface of the hull, I was pleased to find no evidence of corrosion of the steel and no disruption, spalling or cracking of the concrete that would result from corrosion of the steel. In the construction of this

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[1] Inspecting Engineer, Erie Railroad Co., Jersey City, N. J.

Mr. Walter. ship, Celite was used to improve the workability of the concrete. For the information of such as may be interested, I think we have, in this concrete ship, which has been in sea water for more than eight years, an example of splendid assurance that Celite can be used safely.

Mr. Freeman. MR. P. J. FREEMAN.[1]—The matter of hauling over distances was mentioned by Mr. Doe and Mr. Abrams. We have had experience in placing about 40,000 yards of concrete from central mixing plants, using drop-bottom trucks and ordinary end-dump trucks. There does not seem to be a definite relation between the time of hauling and the condition of the concrete. The roughness of the road is also a factor. We hauled concrete for a bridge floor about nine miles over a rough road on which, once in a while, the trucks would be an hour and fifteen minutes on the road. This was just beyond the satisfactory limit. If the trucks got there in fifty minutes, the concrete appeared to be quite satisfactory. If the trucks arrived in an hour and fifteen minutes, they frequently carried considerable water on top of the concrete, and it was rather hard to get the concrete out of the truck. On most jobs we had practically no difficulty at all in dropping it either from the drop-bottom truck or the regular dump truck.

Mr. Bragg. MR. J. G. BRAGG.[2]—In 1917 I had some small part in the experimental work being done on concrete ships. As I recall it, that concrete was designed for a compressive strength at 28 days of 4000 lb. per sq. in., so it might be well to keep in mind that this concrete also had some cement in it. The usual concrete considered, when we investigate failures and that sort of thing, is designed for a minimum strength of 2000 lb. per sq. in. or something like that.

Mr. Ferguson. MR. LEWIS R. FERGUSON[3] (*by letter*).—In the construction of the concrete ships built by the United States Government during the War, it was found that the thin sections with the very large amounts of steel necessary in the design, prevented the filling of the forms with concrete in the ordinary manner. This difficulty was increased, due to the use of harsh aggregate consisting of broken particles of burnt clay. In many places, the reinforcement so completely obstructed the forms that it was impossible to employ either rodding or spading, and yet it was absolutely essential that the forms be completely filled, not only from the strength standpoint, but also because the structure had to be water-tight.

A successful method of accomplishing this object was perfected through the use of light air-hammers. As the concrete was being

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[1] Chief Engineer, Bureau of Tests and Specifications, Department of Public Works, Allegheny County, Pittsburgh, Pa.

[2] Technical Service Engineer, Alpha Portland Cement Co., Trenton, N. J.

[3] Vice-President and Manager, Louisiana Portland Cement Co., New Orleans, La.

Mr. Ferguson. poured, the forms were thoroughly vibrated by numerous hammers adjacent to the locality where the concrete was being placed. The hammers impinged mostly on the outside of the forms, but a few were used on the inside also. In cases where a deep form was being filled, the hammer used on the inside was provided with a long shaft, and in all cases the ends of the hammer shafts were flattened in a mushroom shape about $1\frac{1}{2}$ or 2 in. in diameter. This vibrating method permitted the thorough filling of the forms without difficulty, and produced a very dense concrete. As the wet concrete was subjected to vibration, air bubbles could be seen escaping from it, and when the forms were removed almost no patching work was required.

Careful investigations made a year after the ships built by the writer were launched, disclosed that no seepage at all had occurred. In fact, the concrete in the bottom of the vessel was quite dry, and when the surface was wiped with the hand dust was removed. In the construction of two of our ships, we made a joint where the side met the top of the bilge turn. The reinforcement steel was permitted to extend upward into the adjoining section, and the upper surface of the bottom slab was scrubbed and cleaned with a wire brush before the side sections were poured. Immediately before continuing with the pouring, the surface of the hard concrete was covered with thin grout. No other treatment was used. This joint proved to be absolutely water-tight, and we attribute this condition to the density of the concrete secured by vibration.

It is felt by the writer that in the construction of water-tanks or other structures, which essentially must be water-tight, the vibration method used in the construction of concrete ships might be advantageously employed.

Mr. Gilkey. MR. GILKEY (*presented in written form*).—In certain kinds of work, admixtures probably have their place. The most valuable effect of an admixture is to teach the concrete operator to use stiffer mixtures. He is surprised to discover that a 3-in. slump using the admixture gives less separation and places as easily as a slump of 6 or 7 in. without the admixture. He ascribes the improvement to the magic of the admixture. The strength will be equal to or slightly above that for the same water content (but several inches more slump). In many instances he will find that if he omits the admixture and also leaves out enough water to stiffen his mix to a 3-in. slump, the operator will have a workability differing little from that attained with the admixture and a considerably increased strength by virtue of the lowered water-cement ratio. In the case of a fairly harsh mix, the admixture undoubtedly serves an added function and it may help in water tight-

Mr. Gilkey. ness. If the use of an admixture is the only device that will cause a contractor to cut down the excess fluidity of his concrete, let him be encouraged to use the admixture, for excess fluidity is most objectionable on account of the segregation and laitance that it causes.

Mr. Chapman mentioned the matter of vibrating the forms during or just after placement of the concrete. This is a treatment that should be much more generally employed. A wooden plug inserted in the end of a pneumatic or electric chipping tool will furnish a very satisfactory vibratory action and will insure good concrete and smooth surfaces with mixtures that are usually very difficult to place. This vibration probably has no magic strength-giving effect above that secured by thorough, adequate tamping, but it is a most efficient method of tamping. With wet mixtures the method may actually be harmful in the same manner as excess tamping will increase the segregation, water gain and corresponding laitance.

Mr. Doe. MR. N. L. DOE[1] (*author's closure by letter*).—This discussion has served to emphasize, in an important way, some of the prominent features in connection with the conveying and placing of concrete.

The question of admixtures as discussed by Mr. Walter brings up an important point which was perhaps not brought out as clearly as it should have been, either in the paper, or in discussion, and that is the fact that certain mixes and aggregates are benefited to a much greater extent by admixtures than others. Admixtures benefit a concrete made of coarse aggregates in which very fine materials are lacking much more than they benefit concretes composed of a more normal mixture having a reasonable percentage of fines. It is readily seen, therefore, that some concretes in which the fines are present in an excessive quantity will probably not be benefited by the addition of the admixture, but the quality of the concrete may suffer from too large a proportion of fine inert materials. This same condition results if too large a percentage of admixture is added to any concrete. Very rich mixtures, such as concrete ships are composed of, are benefited less than the poorer mixtures, as the high percentage of cement used is sufficient in itself to fill all of the smaller voids which in the leaner mixes would be filled by the admixture.

In connection with Mr. Gilkey's discussion, there is another feature of admixtures which should be noted. That is the fact that if Celite is added to the concrete, it will absorb a very considerable proportion of the water which otherwise would be used in hydrating cement. If the percentage of Celite is a normal percentage of 3 to 4 per cent, it quite often happens that additional water must be used in order

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[1] Turner Construction Co., New York City.

Mr. Doe. to bring the workability to such a point that the concrete can readily be handled. Mr. Gilkey's statement that the 3-in. slump gives less separation and can be placed as easily as concrete having a slump of 6 or 7 in. may be true for certain special mixes, but it does not seem to apply in general.

Mr. G. E. Warren's discussion[1] appears especially well considered inasmuch as it brings out the fact that the selection of the proper mix is only the beginning of the field control of concrete. Engineers in general are already realizing this and since the new theories of design of mixes has come into general practice, are paying much more attention to the equipment used and the methods of handling the concrete from the mixer to the forms. Mr. Warren has especially emphasized the fact that constant supervision is essential to insure a complete filling of the forms and the incorporation of the concrete around the reinforcement. This, in reality, explains the secret of properly placing the concrete. It is not sufficient to know that any one step is correct; it must be viewed as one operation from the mixer to the forms and as the concrete goes into the forms, it must be of proper consistency without segregation.

DISCUSSION ON CONCRETING IN COLD WEATHER

Mr. Freeman. MR. FREEMAN.—In discussing the quality of concrete in cold weather Mr. McGraw mentions the fact that the concrete should not be permitted to reach a temperature below 50° F. and with that I heartily agree. He mentions that the aggregates should be heated, but I think we ought to put in a word of warning about his reference to the use of common salt (sodium chloride) and calcium chloride, which rather leaves the impression that the use of these would be a satisfactory procedure without heating the materials. Certainly I would never permit it on any work for which I was responsible. The materials would have to be heated, whether calcium chloride were used or not, and I think there ought to be a little word of warning to that effect.

Mr. Gonnerman. MR. H. F. GONNERMAN.[2]—In general, I agree with Mr. Freeman's remarks concerning the use of chemical admixtures when the temperature is below the freezing point of water. Our laboratory has made a great many tests of concrete containing admixtures of sodium chloride (common salt) and of calcium chloride dissolved in the mixing water. These chemicals, when used in the proportions generally recommended for concrete mixtures, lower the freezing point of the

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[1] See p. 433.— ED.

[2] Manager, Research Laboratory, Portland Cement Association, Chicago, Ill.

Mr. Gonnerman. mixing water only a few degrees and should not be depended upon to prevent the concrete from freezing, but rather to hasten its setting and hardening. The use of sodium chloride for the purpose of accelerating the setting process and lowering the freezing point of the mixing water during cold weather is not recommended; 5 per cent of sodium chloride lowers the freezing point of water about 6° F. but reduces the strength of the concrete about 30 per cent.

Mr. Tagge. MR. A. C. TAGGE.[1]—Mr. Freeman and Mr. Gonnerman agreed in warning against the use of chemicals in connection with placing concrete in cold weather or under low temperature conditions. It is not quite clear to me whether their warning is general against the use of all chemicals or merely against the use of chemicals as a substitute for heating. In the district in which I am particularly and most nearly concerned, there has been, as I think generally over the country, a tendency to continue concrete work throughout the winter. A great deal of reinforced concrete work has been put up in cold weather, and it has been a very common practice there for the contractor to add about 2 per cent of calcium chloride to the cement, in the belief that he not only thus helps himself by getting a slightly lower freezing point, but also accelerates the hardening of the cement and makes it necessary for him to heat the structure for a shorter period of time. It was not quite clear to me from the discussion whether that practice was condemned in itself or merely the practice of using chemicals as a substitute for heating.

Mr. Gonnerman. MR. GONNERMAN.—I had in mind the practice of using chemical admixtures as a substitute for heating. There would be no objection to the use of about 2 per cent calcium chloride by weight of cement dissolved in the mixing water in order to accelerate the hardening of the concrete provided heating of the materials, and the concrete after placing was not dispensed with.

DISCUSSION ON TRANSVERSE TESTING OF CONCRETE

Mr. Bragg. MR. BRAGG.—In reference to Mr. Mattimore's paper, my experience in highway work would indicate that the transverse test which he describes is a better measure of the probable behavior of concrete pavement than is the compression test; that is, it is far more indicative of what one will probably find in service than is the compression test.

Mr. Gonnerman. MR. GONNERMAN (*presented in written form*).—Mr. Mattimore has presented an interesting paper which is particularly timely now when there is a demand for a more serviceable and more easily made field test of pavement concrete than the compression test.

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[1] Vice-President and Assistant General Manager, Canada Cement Co., Ltd., Montreal, P. Q., Canada.

TABLE I.—COMPARISON OF UNIFORMITY OF TRANSVERSE AND COMPRESSION TESTS OF CONCRETE.

Transverse tests of 7 by 10 by 38-in. concrete beams.

Compression tests of 6 by 12-in. concrete cylinders.

Mix 1:4 by volume.

Machine-mixed concrete.

Aggregate: sand and pebbles from Elgin, Ill., graded 0–1½ in. (F.M. = 5.50).

Portland cement, a mixture of equal parts of 4 brands purchased in Chicago.

Consistency determined by means of flow table using fifteen ½-in. drops in about 10 seconds. Flow 190; relative consistency about 1.10; nominal water-cement ratio 0.80; net water-cement ratio 0.75.

Concrete mixed in a 3½-cu-ft. mixer for 2 minutes.

Age at test 28 days.

	Modulus of Rupture, lb. per sq. in.					Compressive Strength, lb. per sq. in.				
	3 days	7 days	28 days	3 mos.	1 year	3 days	7 days	28 days	3 mos.	1 year
CANTILEVER LOADING; OVERHANG 30 IN.										
Specimen No. 1	415	665	750	1010	1050	2010	....	5980	7540	8350
Specimen No. 2	360	530	820	965	1180	2060	3240	6020	6950	7300
Specimen No. 3	405	585	745	955	1040	1870	2900	5550	6790	7650
Specimen No. 4	380	535	785	1170	1090	1860	3020	5060	6660	8550
Specimen No. 5	375	570	855	940	1120	1840	3120	5400	7530	8740
Average	385	575	790	1010	1095	1930	3070	5600	7090	8120
High	415	665	855	1170	1180	2060	3240	6020	7540	8740
Low	360	530	745	940	1040	1840	2900	5060	6660	7300
Difference, per cent of Low	15.3	25.5	14.8	24.5	13.5	12.0	11.7	19.0	13.2	19.7
Difference, per cent of Average	14.3	23.5	13.9	22.8	12.8	11.4	11.1	17.2	12.4	17.7
Mean Variation from Average, per cent	4.7	6.7	4.7	6.5	4.1	4.5	3.6	5.7	5.0	6.3
CENTER LOADING; SPAN 36 IN.										
Specimen No. 1	490	625	870	885	1155	1840	3500	5740	6670	7700
Specimen No. 2	420	615	790	945	1235	1840	3390	5630	6050	7950
Specimen No. 3	460	615	805	925	1085	1990	3050	5590	7100	7300
Specimen No. 4	465	630	750	765	1135	1880	3320	5700	7020	7860
Specimen No. 5	460	550	805	905	1155	2040	3060	5590	6250	7420
Average	460	605	805	885	1155	1920	3260	5650	6620	7650
High	490	630	870	945	1235	2040	3500	5740	7100	7950
Low	420	550	750	765	1085	1840	3050	5590	6050	7300
Difference, per cent of Low	16.7	14.6	16.0	23.5	13.8	10.9	14.7	2.7	17.4	8.9
Difference, per cent of Average	15.2	13.2	14.9	20.3	13.0	10.4	13.8	2.7	15.9	8.5
Mean Variation from Average, per cent	3.3	4.0	3.4	5.4	2.9	4.1	5.1	1.0	5.6	3.0
THIRD POINT LOADING; SPAN 36 IN.										
Specimen No. 1	390	565	725	890	840	2240	3420	5660	6920	8710
Specimen No. 2	420	560	735	815	935	2130	3240	5850	6700	7240
Specimen No. 3	385	545	705	800	970	16 0	2960	5950	6920	8090
Specimen No. 4	260	550	680	870	1025	1830	3030	5220	7430	7450
Specimen No. 5	390	510	655	740	1095	2060	2780	5450	6170	8450
Average	370	545	700	825	975	1980	3030	5630	6830	7990
High	420	565	735	890	1095	2240	3420	5950	7430	8710
Low	260	510	655	740	840	1620	2780	5220	6170	7240
Difference, per cent of Low	61.5	10.8	12.2	20.3	30.4	38.3	23.0	14.0	20.4	20.3
Difference, per cent of Average	43.2	10.1	11.4	18.2	26.3	31.3	21.1	13.0	18.5	18.4
Mean Variation from Average, per cent	11.6	2.8	3.7	5.6	7.2	10.1	5.1	4.1	4.6	6.4
Grand Average, Mean Variation, per cent	6.5	4.5	3.9	5.8	4.7	6.6	4.6	3.6	5.1	5.2

Mr. Gonnerman.

He has made the deduction that since the modulus of rupture is used as the basis of design of rigid pavements the transverse test is the logical one to use in determining the quality of pavement concrete in the field. It is a relatively inexpensive test and he concludes from tests which he has made that it gives concordant results.

During the past year our laboratory has made an extensive series of experiments comparing the transverse with the compression test, the specimens being made from identical mixtures cured in the same manner and tested at the same age. Some data from these tests are shown in Table I.

These tests bear out Mr. Mattimore's conclusion that the transverse test is reliable and gives very satisfactory results. In our tests, we used five specimens in a set made on different days and we found about the same mean variations as shown by Mr. Mattimore's tests. It was found, however, that there was just as good agreement between the compression specimens as was obtained in the transverse tests. This does not quite agree with Mr. Mattimore's results in which he found a somewhat wider spread in the case of the compression specimens.

In our experiments, we used third point and center loading in addition to the cantilever method. The same values of modulus of rupture were not found for all methods of loading although the differences between the values for center and cantilever loading were not great. For all ages, the values for cantilever loading were slightly below those for center loading while those for third point loading were about 70 lb. per sq. in. lower than the average of the values for the other two methods at 3, 7 and 28 days and about 140 lb. per sq. in. lower at 3 months and 1 year. The lower moduli of rupture for third-point loading are probably due to the fact that with cantilever and center loading the maximum bending moment is developed at only one section of the beam, over the support and under the center load, respectively, whereas with third point loading the maximum moment extends over the middle third of the beam so that with this method the conditions for locating the weakest section of the specimen are more favorable than is the case with the other two methods of loading.

In the event that the transverse test is generally adopted for field control, it will still be desirable to make occasional check tests on compression specimens in order to correlate the results with the experience of the past which is largely recorded in terms of compressive strength.

In this connection, it is of interest to call attention to another feature of Mr. Mattimore's paper. In his reference to tests from

Bulletin 11 of the Structural Materials Research Laboratory, he quotes data to show that the modulus of rupture increases with the fineness modulus of the aggregate beyond the point where the compressive strength has begun to fall off. This same tendency was noted also in another series of tests made in our laboratory during the past year. These results would seem to indicate that some caution is necessary in the use of the transverse test as a sole criterion of concrete quality as it might result in the use of mixtures having a sand content too low for watertight and durable concrete. **Mr. Gonnerman.**

MR. H. S. MATTIMORE.[1]—In comparing the data and reports on molded *versus* drilled cores, one fact that I believe should be brought out is that in the Slater and Walker tests the drilled cores were taken from premolded beams or slabs. Mr. Walker, who is present, could inform us relative to this. **Mr. Mattimore.**

MR. STANTON WALKER.[2]—The cores to which Mr. Mattimore refers were drilled from slabs cured under the same conditions as the structure up to about 28 days, when the cores were cut, and in the air of the laboratory thereafter. At Camden, 9 slabs 3 by 5 ft. by 8 in. were made; 3 on each of three floors. Nine cores, 4½ in. in diameter, were cut from each slab at about 28 days to be tested, 3 at 28 days, 3 at 3 months, and 3 at one year. At Newark Bay 6 slabs were made, 12 in. thick instead of 8 in.; 6⅜-in. cores were drilled from them. In addition to the slabs, at Camden two test columns were cast, each 10 ft. long; one of them was 12 in. square and the other, 12 in. in diameter. These columns were cast on the structure and after about 28 days were sawed into four test specimens each 24 in. long. Standard 6 by 12 in. cylinders were molded from the same batches as those from which the slabs and columns were made. The cylinders were cured in damp sand. **Mr. Walker.**

The 28-day tests at Camden and the 3-month tests at Newark Bay gave about the same strength for the cores as for the companion cylinders. At 3 months the Camden cores gave about 14 per cent less strength than the cylinders. After 1 year the strength of the cores was about 26 per cent less than the cylinders. These differences may be accounted for by the differences in curing conditions.

The specimens sawed from the columns showed lower strengths than the cylinders molded from the same batch. The strengths of these specimens were greatest for those sawed from the bottom of the columns, and decreased more or less uniformly as they were sawed from nearer the top.

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[1] Engineer of Materials, Pennsylvania State Highway Department, Harrisburg, Pa.

[2] Director, Engineering and Research Division, National Sand and Gravel Association, Washington, D. C.

Mr. Walker. Complete details of these tests up to the 3-month period may be found in the report on "Field Tests of Concrete Used on Construction Work" by W. A. Slater and Stanton Walker, in the Proceedings of the American Society of Civil Engineers for January, 1925.

Mr. Mattimore. MR. MATTIMORE.—This is just the factor I wish to bring out. The curing of a large mass, which we might term curing in place, may be somewhat different from the curing of precast slabs or beams.

Mr. Teller. MR. L. W. TELLER[1] (*presented in written form*).—As Mr. Mattimore has pointed out, it is logical to develop a test which measures the modulus of rupture of pavement concrete, since this is the generally accepted basis for the design of pavements which employ this material.

That this is realized by engineers in the highway field is evidenced by the fact that nearly all of the state and other laboratories have adopted some scheme of beam test for measuring the modulus of rupture. These schemes vary widely both in the form of the devices used and in the test procedure. Some of them, such as the one described in Mr. Mattimore's paper, are extremely simple; others are more complicated. The method of supporting the specimen varies from a condition of almost complete restraint to one which is practically that of a simply supported beam. The method of applying the load varies. Many of the devices use shot, water or sand flowing at a more or less constant rate into a container on the end of the extension arm. Others use sand bags or lead weights laid on a platform; still others apply the load through a spring dynamometer.

This variety suggests that there is a very urgent need for data which will furnish a basis for some standardization of this test, particularly with regard to (1) method of restraining the specimen, and (2) method and rate of load application. The author says that this subject is being studied by committees, but this takes time and so far there has been but little published.

In the laboratory of the Bureau of Public Roads at Arlington, some attempt is being made to study this test, and, although the data thus far obtained are somewhat meager, they are being presented with the hope that they will be of value to others who are making an effort to standardize the transverse test.

The tests were all made on 6 by 6 by 30-in. beams of 1:2:3½ proportions, 28 days old. Great care was exercised in fabrication and curing to secure uniformity. In the first series of tests, one brand of cement and one type of coarse aggregate were used; in the second series, both the brand of cement and the type of aggregate were changed.

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[1] Engineer of Tests, U. S. Bureau of Public Roads, Washington, D. C.

Mr. Teller. Three methods of restraining the specimen in the cantilever test were employed and simply supported beams were also tested with a centrally applied load. In the cantilever test, lead shot was allowed to flow into a container on the end of the beam at a rate of 100 lb. per minute, except for a few tests in which rates of 50 and 200 lb. were used.

Method 1.—This is the procedure which has been adopted in our laboratory for lack of a standard. The specimen rests on a flat machined steel plate. On the upper surface of the beam, a sheet of ¼-in. rubber packing is placed. Over this is a ⅝-in. steel plate which is pressed against the rubber by two transverse steel straps held down by bolts anchored in the bed of the testing machine. The extension arm is very similar to that described by Mr. Mattimore, but, as previously remarked, lead shot flows into a container on the end of the arm to apply the load.

Method 2.—This method was considered as being the simplest and for this reason it has often been used in the field. The specimen rests on the steel bed plate, as in Method 1, but is restrained only by a single transverse steel strap across the rear end of the specimen.

Method 3.—A ½-in. round steel rod was placed transversely under the beam at the edge of the bed plate. Under the rear end of the beam a ½ by 1-in. flat steel strip was placed transversely. The beam was held down by a steel strap placed across the rear end over this support. It has been claimed by some that such an arrangement leads to more consistent results.

Method 4.—The specimens were supported on two rocker supports 28 in. apart and the load applied at the mid-span. These tests were made in a testing machine, operated by a hand wheel.

The data secured from this first series of tests is given in the following table:

Method	Number of Tests	Average Modulus of Rupture, lb. per sq. in.	Relation to Method 1, per cent
1	6	436	100
2	6	427	98
3	6	440	101
4	5	495	112

USING METHOD 1 AND VARYING THE RATE OF APPLICATION OF LOAD

Rate	Number of Tests	Average Modulus of Rupture, lb. per sq. in.	Relation to 100 lb. per Minute Rate, per cent
50 lb. per minute	6	426	98
100 lb. per minute	6	436	100
200 lb. per minute	6	474	109

Mr. Teller. These data showed some promise but it was felt that a greater number of specimens was necessary to study not only the magnitude of the modulus of rupture values but the relative consistency of tests by the different methods. Twenty-eight-day tests on a second series of beams have just been concluded in which methods 1, 2, 3 and 4 were again compared. These data are summarized in the following table:

Method	Number of Tests	Average Modulus of Rupture, lb. per sq. in.	Relation to Method 1, per cent	Maximum Variation		Mean Variation		Standard Deviation	
				lb. per sq. in.	per cent	lb. per sq. in.	per cent	lb. per sq. in.	per cent
1.........	24	536	100	50	9.3	18	3.4	22	4.2
2.........	24	543	101	75	13.8	26	4.7	33	6.1
3.........	24	567	106	52	9.2	22	3.9	28	5.0
4.........	12	601	112	87	14.5	35	5.8	47	7.8

It is indicated by these data that, of the four methods investigated, for consistency method 1 is the best; that method 3 is better than method 2 and that method 4 is the least consistent. From the standpoint of magnitude of the modulus value obtained, both the first and second series indicate no great difference so long as the cantilever test is used although method 3 seems to give slightly higher values. When the beam is tested on simple supports, however, considerably higher values were obtained in both series of tests. With this method of test, it is difficult to be sure that no horizontal component is being developed at the reactions and this may explain why the values obtained by method 4 are 112 per cent of those found by method 1.

The few data on the effect of rate of load application indicate that the rate should be standardized. A convenient rate would be one which developed the maximum fiber stress at a rate of 200 lb. per sq. in. per minute. Perhaps other tests will indicate a more suitable value.

Mr. Crum. MR. CRUM.—I cannot refrain from disagreeing with Mr. Mattimore and Mr. Teller on the point that compressive stresses in concrete pavement are not worthy of consideration in the design of a slab. I drove over hundreds of miles of concrete pavement on the way down here; I passed a good many examples of structural failure due to compressive stresses that were undoubtedly set up by temperature or moisture changes, and I do not think that concrete slabs for pavements are properly designed unless the compressive stresses due to temperature and moisture changes are taken into account. I have found the transverse tests to be very valuable in connection with concrete pavement work. I have also found compressive tests to be of great value in research work connected with concrete roads, and I am of the opinion that the transverse test should not be considered a substitute for compressive tests, but merely an additional tool to be used with them to help us in our work of getting these structures properly built. **Mr. Crum.**

MR. D. A. ABRAMS.[1]—I should like to lend my voice to the sentiment expressed by Mr. Crum. It seems to me that we should be very slow to accept the conclusion that the transverse test is better than the compression test. In any test of this kind, non-uniformity simply reflects the non-uniformity of the materials in the specimen and of our technique in making and testing them. I see no reason why, if all things are controlled properly, we should get any different variation in the compression test from what we find in the transverse test. I do not mean to say that the transverse test is not proper, but I would be very much surprised if we do not find on a more thorough examination, quite a close correlation between compression and transverse tests. It will not always be the same value, but the relation will be consistent, it will follow certain laws; it may be slightly different for rich mixtures and for lean ones, or other factors of that kind, but if the quantities are controlled and the conditions uniform, one test should produce just as uniform results as the other. **Mr. Abrams.**

There is frequently a tendency to cut corners in making concrete tests. It is difficult to get the engineer to realize that some of the apparently insignificant things are of great importance so far as the uniformity and the value of tests are concerned. The mere matter of capping the compression test specimen is of extreme importance and one which in too many cases is neglected. When one realizes that a non-uniformity of only a few hundredths of an inch in the loading surface of compression specimens makes an enormous difference in the strength, one will see how important proper capping is. There is no better way to prepare the ends of the test cylinders than by using machined plates. Very few laboratories do that; some of our best laboratories are making the mistake of using sheared steel plates in molding test cylinders. In general sheared plates are not plane and are far inferior to machined plates of proper thickness. These factors do not affect the transverse test, particularly if one provides a cushion for the load. Improper test procedure is more likely to penalize the compression tests and is responsible for many erroneous conclusions.

MR. FREEMAN.—I think we are overlooking one of the essential things that the transverse test gives us. Tying directly back to the design of the road slab we are able to open intersections of highways **Mr. Freeman.**

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[1] Director of Research, International Cement Corporation, New York City.

Mr. Freeman. at very much shorter periods than we have been accustomed to do in the past. No doubt we could do the same thing with the compression test but we have not been doing it. By using the chart on which Mr. Mattimore showed the various moduli of rupture, we have been opening sections of bridge floors, bridge approaches and road intersections at perhaps not longer than 5 days where we used to keep them closed for 21 days. Perhaps we used calcium chloride and heat or maybe extra cement. The main object was to get the job open by having a machine out in the field which could be operated by the field engineer, with which he could obtain the information necessary to open that road for traffic at the earliest possible minute. I do not think we brought that feature out at all, regardless of the fact that the test may or may not be as accurate as the compression test.

Mr. Bragg. Mr. Bragg.—If I had not been sold on this test for pavement work, Mr. Abrams would have sold me. I think the reason why one has trouble in getting concordant results in compression tests is due to the fact that there are so many variables in the compression tests that will affect the strength. Many of those are eliminated in this transverse test, and I think that is one reason why we get better results with this test. However, I agree with Mr. Crum and Mr. Abrams in that I do not think the compression test should be dropped; I think it is very important, but in comparing the details of the crack survey made on 450 miles of hard surface road in New Jersey, the indications certainly are that the transverse tests will give a better indication of how the concrete is going to behave in actual practice than the compression test. We do have roads showing a high compressive strength that are not standing up as well as some with low compressive strength, and the transverse tests indicate more nearly how the pavement concrete is actually going to behave in the field.

Mr. Kellam. Mr. Fred Kellam.[1]—I do not believe that either Mr. Crum or Mr. Abrams was trying to decry the value of the transverse test, but they were pointing out the danger of using the transverse test as the only test. I had a little experience which called attention to the danger of concentrating all our attention upon the particular feature in design which is giving most trouble at the time. We realized that the transverse strength is now being primarily used as the basis for pavement design. That is not the only feature that enters into the value of pavements. Mr. Freeman has mentioned the fact that we are using that test as a means to obtain the valuable end of opening pavements earlier to traffic; but we are going ahead on the assumption that if we take care of the transverse strength the other qualities of the concrete will run parallel. Under normal conditions that may be true, but if we pursue that end without paying attention to any of the others, we are likely to get into trouble. Some months ago, in the Indiana State Highway Laboratory, we gave some attention to the ball test developed at Purdue University on the theory that surface hardness was an easy thing to measure, and if, as it appeared, it was parallel to the other qualities of concrete, that would be a splendid way to measure the properties of concrete in pavements. We worked up a portable testing device on that principle by using energy developed from a dropped weight and we got along fine in the preliminary investigation, until we developed the fact that moisture affected the surface hardness entirely out of proportion to its effect upon other properties of the concrete. In fact, by this test we learned that a piece of pavement could have been opened to traffic very safely until we threw some water on it and then in two or three hours it was in a much worse condition than it had been several days previously. I believe that that is worthy of some investigation. We have assumed that surface hardness need not worry us. We used to worry about the wearing of concrete pavements. We assume now that if we take care of compressive strength, or latterly of transverse strength, satisfactory surface hardness will follow without attention. I believe that it should be given some attention and all the elements entering into the service of a concrete road or any other structure should be watched, at least out of the corner of our eye, while we proceed with the main issue of transverse or compressive strength. **Mr. Kellam.**

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[1] Assistant Chief Engineer, Indiana State Highway Commission, Indianapolis, Ind.

MR. MATTIMORE.—Mr. Abrams brought out in his discussion some points which to me are excellent arguments for the use of transverse tests. In all our testing work, and especially so in our field testing, we desire simplicity, providing we secure accurate and concordant results. I believe there are enough data in my paper to demonstrate the simplicity of this transverse test, also data indicating that more concordant results are secured with it than with the compression test as usually carried on in field testing in most laboratories. **Mr. Mattimore.**

I did not wish to give the impression that data on compressive strength of concrete are not of value and necessary, but if a relation can be determined between transverse and compression tests, why not use the transverse test due to its simplicity and the concordant results obtained and convert the figures so obtained to compressive strength, rather than use the complicated and more variable test to ascertain the transverse strength?

Mr. Abrams.

MR. ABRAMS.—I am not questioning the transverse test; my point was that I think we are likely to make a mistake if we conclude that the transverse test *per se* is any better than the compression test. I do not believe it is. It may be an easier test to make in the field, but I do not believe that it is essentially better. I would be very much surprised if one test did not give just as good a measure of concrete quality as the other, providing both were made under equally satisfactory conditions. That is my whole point. I do not mean to say that the transverse test is not a valuable indication.

As to the point made by Mr. Freeman, I fully agree that concrete roads have in the past been kept closed to traffic too long. Most concrete roads could be opened, if properly handled, in 4 to 10 days, whereas they are generally closed for 3 to 4 weeks. The mere fact that the transverse test has pointed that out does not mean that the same information might not have been gotten from the compression test or by other means.

DISCUSSION ON FIELD TESTING

Mr. Gilkey.

MR. GILKEY (*presented in written form*)—Under the general heading "The Sample," Mr. Young sets forth the divergent points of view relative to the function that the sample is to serve. Is it to be only a check upon the quality of the concrete that went from the mixer to the forms, or is it to represent the concrete of the structure as influenced by the mixture used and all subsequent treatment? On large and important work both sets of information are important and they should not be confused. Mr. Young points out the difficulty in obtaining samples truly representative of the structure because of unequal temperature and drying effects. The "job" control should only be attempted by experienced operators who have adequate facilities. The quality control specimens should be the usual ones taken. These will be obtained in the standard manner, moist cured, and tested wet, regardless of the treatment given to the structure. Mr. A. R. Lord[1] makes this distinction very clearly. The writer touches upon the subject somewhat further in his discussion of Mr. Lord's paper.

Relation of Field Tests to the Strength of the Concrete.—Under this heading at the close of Mr. Young's paper, it is surprising to see one of his broad experience and usual keen discernment taking such a neutral stand. So far as the writer has been able to detect, in all instances in which job samples (whether drilled cores, or specimens job-cured) were stronger than samples moist-cured in standard laboratory fashion, there have been present factors that invalidated the comparison. **Mr. Gilkey.**

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[1] Notes on Wacker Drive, *Proceedings*, Am. Concrete Inst., 1927.

It is probable that laboratory-cured specimens will always equal or exceed the strengths of job-cured specimens providing the temperature of the job-cured specimens is not higher than that of the laboratory specimens. Even the extra temperature would be against the job specimen or drilled core if ample moisture were not also present. Below are listed some factors that invalidate many such attempted comparisons:

1. Strengths of dry cores are compared with strengths of moist laboratory-cured specimens.
2. Relatively shallow cores are compared with laboratory specimens with no attempt to compensate for the divergency in the relation of height to diameter.

Dry concrete has a strength from 10 to 40 per cent above that of the same concrete when moist. Short stocky specimens may have a compressive strength several times that of the standard cylinder with height double the diameter.

The references cited in support of the core giving excess strength are typical of many such citations that could be given.

The tests by R. E. Goodwin[1] were unusually thorough for that time, but that was before the significance of the water-cement ratio was appreciated and there were so many other factors of which little was known that results from that period are of scant value as evidence in the present instance. Moreover Mr. Goodwin cites his unit weights as 147 and 151 lb. per cu. ft., respectively, for the sawed-out prisms and laboratory-cured specimens. These unit weights are evidence that the concretes were not similar or else that the higher-strength sawed samples were drier at test. This is the reasonable explanation as the difference is a very representative one for similar concretes of different moisture contents. His strength differentials are well within the range that moisture difference will account for.

Mr. Young's last reference, J. G. Bragg, *Engineering News Record*, June 12, 1924, p. 1012, was probably alluding to Mr. Mattimore's results[2] in the latter part of the following citation. The fore part is applicable to the general problem. Mr. Bragg states:

> "The question of considering all variables and obtaining fair averages is particularly stressed because of the fact that since the advent of the core drill certain articles have been published in which some rather absurd conclusions were drawn from results which could not possibly be comparable. In most instances either the age of the tested concrete varied throughout the experi-

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[1] *Engineering Record*, September 4, 1915.

[2] *Engineering News-Record*, January 12, 1922, p. 73.

Mr. Gilkey. ment, no corrections were made for the form of the specimen used or too many projects were included with an insufficient number of specimens taken on each project. One writer remarks the astonishing fact that cores cut from the finished pavement showed much higher strengths than did the control specimens made when the concrete was placed. In view of the fact that his control specimens were 6 by 12-in. cylinders tested at 28 days, some of the 6-in. cores cut were two years old with a height of 6 to 8 in., and no correction made on the results for height to diameter ratio, it would indeed have been remarkable had he not experienced higher strengths in the field-cut cores."

Mr. Bragg does not specifically mention the matter of differences in moisture content which are also important.

The writer recently had occasion to take rather strenuous exception to test results that made indiscriminate comparisons of concrete in all stages of dryness and which led to some very absurd conclusions.[1]

In Mr. Young's other citation, Mr. Gage probably despaired of any agreement between cores and molded samples because of such unrecognized variables as those mentioned. Slater and Walker's findings are probably sound.

It is very difficult to bring concrete specimens to any uniform degree of dryness. From 10 to 24 hours' immersion will produce practical saturation and it is therefore advisable that all strength comparisons be made on saturated specimens.

For pavement concrete the saturated strength is the only strength that can safely be counted upon. In the interior of buildings the dry strength is the one invariably present but if drilled or sawed specimens are to be compared with moist-cured specimens, they should be immersed a day prior to test or the comparison will be entirely misleading as to relative strengths and unit weights.

Perhaps the last of Mr. Young's paper seems to have received more than its proper share of comment, but this is a point of real importance and so commonly misunderstood that any amount of over emphasis that may be necessary to put the point across is justified.

When we see, within the year just passed, a very large and costly state highway investigational project performed in cooperation with the Portland Cement Association laboratory and staff made practically worthless because of variable and unevaluated moisture contents in the concrete specimens at test; when we see our own active and capable fellow member, Mr. Mattimore, offering as late as 1922 conclusions from tests of specimens of widely different ages, relative proportions, and probable moisture content, without even an attempt at evaluating the several variables (a thing that could not be done

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[1] *Proceedings*, Am. Soc. Civil Engrs., January, 1927, pp. 79–93.

Mr. Gilkey. any way in such a case); and when we see our clear-visioned Canadian investigator being influenced in his own judgment by the conclusions of Mr. Mattimore based upon strange evidence so obviously misinterpreted, and also by results from tests of a 1915 vintage; is it not time that we listen more critically and less gullibly when our experts are speaking? We do like terse, boiled-down statements of fact in headline form, but we must expect to make many false starts and do much back tracking if we persist in accepting promiscuous (and oftentimes even expert) interpretations of test results and neglect to give the tests themselves a searching scrutiny.

In all mathematical analyses it is essential that there be as many equations as variables. It is equally true in investigational work, that the problem must be isolated by either eliminating all but one major variable or by multiplying the projects until simultaneous values may be found for all. With these points clearly in mind it is often possible to avoid giving undue credence to heavy-looking evidence that is thin or fallacious. In few fields have more well-intentioned but highly misleading deductions been offered. We accept them all and wonder why we find it so difficult to assimilate and digest things concrete. An occasional symposium such as this one should go far toward clearing the atmosphere of the non-essential and focusing our attention upon the relatively few major points that can be accepted, modified or rejected after being subjected to the spot light of constructive criticism.

Mr. Young. MR. R. B. YOUNG[1] (*author's closure by letter*).—Mr. Gilkey criticizes the author's paper on the ground that he has taken a neutral stand on the question of the relation between strength of concrete in a structure and that obtained by field specimens. In view of the meagerness and weakness of the data, as pointed out by Mr. Gilkey, it would seem more proper to congratulate the author on the omission of his personal views, for to have included them in the paper would only have tended to obscure the fact that on this very basic phase of the science of testing concrete our knowledge was very unsatisfactory.

Mr. Gilkey has not left his readers entirely clear as to where he himself stands on this very important question, although one would naturally assume from the tenor of his remarks that he believes the concrete in a structure is not as strong as field tests would indicate. If he has data to support this opinion it would be helpful to others to have him present it to the Society.

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[1] Senior Assistant Laboratory Engineer, Hydro-Electric Power Commission of Ontario, Toronto, Ont., Canada.

Mr. Young. Mr. Gilkey's reference to experts is quite good. I hope he does not hold as low an opinion of them as his remarks would lead one to believe, for whether he classes himself in that category or not, his colleagues quite justly consider him one, so that his remarks are something of a boomerang.

GENERAL DISCUSSION

Mr. Kitts. MR. J. A. KITTS[1] (*by letter*).—This symposium is another step from the haphazard age of making concrete to that of technological concrete manufacture. It gives recognition, in statements throughout the papers, to the fact that concrete is manufactured on the job and that the engineer must not only design the structure, but he must provide for reliable control of the manufacture of the concrete. The authors have brought out many new ideas of importance and are to be commended for the advanced work accomplished.

I have a strong conviction that more emphasis should be made of the fact that concrete production is a manufacturing process; that the manufacture of concrete of quality requires thorough, constant and understanding technical and practical control of the proportioning, measuring, mixing, placing and curing, in coordination with and supported by, cement, aggregate and concrete testing as a daily routine part of such production control. I am also convinced that more attention should be given to other qualities as well as to strength.

Considering the various classes of structures for which concrete is suitable, the qualities demanded of concrete and to which attention should be given are: (1) Economy; (2) durability; (3) strength; (4) density; (5) imperviousness; (6) weight; (7) resistance to fire, ice, wear, chemicals, etc.; and (8) (very important), essential workability, flowability and cohesion of the fresh mix. The relative importance of each depends upon the purpose, but economy is always important. These qualities should be known with practical certainty before the concrete is placed instead of after or not at all, as is the prevailing situation.

There is evidence that these qualities depend upon some or all of the following items of the composition and manufacture of the, concrete:

Cement quality,
Cement content,
Cement to space occupied,
Water-cement ratio,
Quality of aggregates,
Shape of aggregates,

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[1] Consulting Concrete Technologist, Kitts and Tuthill, San Francisco, Calif.

Mr. Kitts.

Maximum size of aggregates,
Coarseness (or fineness modulus) of aggregates,
Surface area of aggregates,
Uniformity of grading of mixed aggregate,
Character of uniformity of grading of mixed aggregate,
Cement to voids in mixed aggregate,
Cement to voids in fine aggregate,
Sand to voids in coarse aggregate,
Mortar to voids in coarse aggregate,
Accuracy of measurement of aggregates, cement and water,
Mixing time and efficiency of mixer,
Temperature of curing,
Moisture of curing,
Efficiency of placing plant, and
Workmanship in mixing, placing and curing.

Economical mixtures, of the required quality in other respects, depend upon the utilization to the fullest possible extent of these conditions which economically benefit that quality, likewise, the best quality within the limitations of the requirements. The various empirical formulas for strength are apparently only of general significance, insure only an approximation of the strength expected, and have no definite bearing on the other qualities. This is shown by the analyses, under the auspices of Committee C-9 on Concrete and Concrete Aggregates, of the Wisconsin, Texas, Ontario, Toronto, and Philadelphia tests. Any value of the one variable of the particular formula[1] gives widely different results. It seems evident that the time is not ripe to predict the strength or other quality of concrete by formula and formulas involving only one variable cannot possibly account for the many variables which affect any particular quality. More research and more complete data of concrete mixtures are required before we can accurately differentiate the various elements which affect the qualities.

The solution of the problem of producing concrete of quality, then, is not in empirical formulas but in thorough technological control of the manufacture, utilizing such knowledge as we have of the physics of concrete, necessarily conducting concrete research as a normal operation of such control, and in the development of a comprehensive concrete technology. Results on California projects of 3000 to 457,000 cubic yards have shown that this is the solution of the problem of producing concrete of quality with economy, and the

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[1] See "A Study of the Data of 'Series 201' and Wisconsin Tests Showing the Relation of the Compressive Strenth of Concrete to the Water-Cement Ratio, Space-Cement Ratio and Grading of the Aggregate," by R. A. Nelson, Appendix I to Report of Committee C-9 on Concrete and Concrete Aggregates, *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I, p. 361 (1927).

Mr. Kitts. requirements of these projects have brought forth many new developments in concrete technology.

These developments and their application are quite pertinent to the subject under discussion and will be outlined briefly. They are founded on the new conception of concrete production, for quality, as a manufacturing process requiring constant technological control. They are designed for manufacturing concrete to economical and definite standards of strength, density, impermeability, weight, durability, and of essential workability, flowability and cohesion of the fresh mix. They make concrete research a normal operation of construction with savings in cost over empirical control, with results sought and with greater uniformity in the required quality of the product. These developments and their application constitute the first economic concrete technology. They are enumerated as follows:

1. A comprehensive physico-mathematics of concrete materials and mixtures. This is the foundation of the testing, proportioning and measuring procedure. It is an application of fundamental physics and mathematics to the measurement and interpretation of the physical properties, relations and characteristics (*a*) of rock particles,—specific gravity, absorption, hardness, fusibility, solubility, etc.; (*b*) of mixtures of particles, density,—gradation of sizes, fineness modulus, surface area, silt, moisture, bulking by loose measure and moisture, and relations of absolute, dry-rodded, loose-measured and inundated volumes and of weights; (*c*) of mortar and concrete mixtures—density, weight, water-cement ratio, consistency, yield, cement content, cement to voids in the mixed aggregate, cement to space occupied, sand to voids and mortar to voids in the coarse aggregate, permeability, predetermined strength at any age, etc.

2. Coordination of the tests for the various physical characteristics of aggregates. By this coordination, the tests of aggregates for density, specific gravity, absorption, moisture, silt, bulking, etc., are made with the least number of operations. It is neither expedient nor economical to test for density, then for specific gravity, then for weight per cubic foot, etc., as the time and labor of handling, measurement and of calculation are increased a number of times over that actually required by a coordinated test procedure.

3. Coordination of aggregate tests and aggregate mixture tests. This reduces the number of test operations required.

4. Coordination and systematization of aggregate, aggregate mixture, mortar and concrete mixture tests. This greatly reduces the test operations required for the determination of the characteristics of a concrete mix, weight, density, water-cement, cement-space,

yield, cement content, granular structure, voids relations, consistency, **Mr. Kitts.** predetermined strength, etc., and expedites concrete testing with economy. It facilitates the maintenance of quality by the expedition with which a complete concrete test may be made.

5. Coordination of field and laboratory measurements by actual, dry-rodded, loose-measured and inundated volume, and by weight. This makes it possible and practicable to accurately duplicate laboratory measurements in the field by weight or volume. It is essential, in laboratory maintenance of quality, that the field measurements of aggregates by loose-moist volume and the corresponding laboratory measurements by weight are identical in absolute volume of particles, likewise by other methods of measurement.

6. An algebraic method of combining any number of sizes of aggregates for any uniform grading. This makes it possible to calculate in a few minutes the proportions of three, four or any number of sizes of aggregates to fit a grading curve or equation, and is more accurate, and much faster than the old graphical cut-and-try method. It is accomplished by employing the fineness modulus principle in connection with a grading equation such as the Talbot-Richart equation:[1] $p = (d/D)^n$

7. The Kitts-Peugh grading equation, for grading of sizes of particles in a mixed aggregate, $r = \frac{1 - (d/D)^m}{1 - (a/D)^m}$, in which r is proportion by absolute volume retained on screen opening of d inches, D and a are maximum and minimum sizes, respectively, and m is an exponent. This provides for uniform grading of any fineness modulus and surface area. It is similar to the Talbot-Richart equation except that it limits the minimum size of the aggregate, excluding silt. A particular value of m provides the mixture of the required quality with the greatest possible economy under the fixed conditions.

8. Proportioning aggregates for uniform grading by size and absolute volume of particles. This is equivalent to grading by size and weight when the specific gravities of the individual aggregates are the same. This is a measure for maintenance of quality based on the idea that the physical character of a concrete mix (of a particular aggregate and cement) is fixed by the absolute volume and gradation of sizes of the aggregate particles and the absolute volumes of cement and water.

9. Coordination of the fundamentals of the various theories of proportioning concrete mixtures. This takes into consideration the

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[1] See "The Strength of Concrete, Its Relation to Cement, Aggregate and Water," by A. N. Talbot and F. E. Richart, *Bulletin No. 137*, Engineering Experiment Station, University of Illinois.

Mr. Kitts. voids theories, that of water-cement ratio, cement to space occupied, cement content, density; and of density, surface area, fineness modulus, and uniformity of grading of the mixed aggregate. It has many advantages over any one-theory proportioning method as it takes advantage of every element which benefits quality and effects economy.

10. Establishment of technological control as a daily routine of concrete production. The man-hours of control are arranged to keep up with the changes in density, specific gravity, moisture and bulking of the individual aggregates, and the modifications of proportions, for maintenance of quality, required by these changes. By this technological control, the physical characteristics of a proposed concrete mix, density, weight, water-cement, workability, minimum probable strength, etc., are determined before the concrete is placed, insuring quality and effecting economy. The effectiveness of this control in the maintenance of quality depends upon items Nos. 1 to 9 and also upon scientific insight into the laws of concrete mixtures, and practical "concrete sense." One man-hour of laboratory control is required for every fifty yards as a maximum under favorable conditions. Control costs vary from 3 to 40 cents per cubic yard, depending upon the production rate and other conditions.

11. Coordination of aggregate and concrete production control on major projects. Technical production control of the aggregates serves to maintain the uniformity and other qualities of the product and the quality of the concrete may by this coordination of production be maintained with better uniformity and with greater economy than is possible by control of concrete production alone.

12. Other practical and scientific elements of aggregate and concrete manufacturing control, specifications, plant layout, apparatus, etc., to maintain the uniformity of quality and reduce the margin of excess cement required.

The system of aggregate and concrete production control, applying these developments, consists of:

1. Maintenance of a central cement, aggregate and concrete testing laboratory at the major structure of the project; field laboratories for moisture tests of aggregates, slump tests of concrete and the making and preliminary curing of test specimens; and an aggregate testing laboratory at the aggregate plant.

2. Preliminary research tests of cements, aggregates, and of aggregate, mortar and concrete mixtures, varying grading, fineness modulus, cement and water content, to determine the most economical mixtures of the required physical properties and workability; determination of strength, density, weight, water-cement ratio, cement-

space ratio, consistency, yield, cement per cubic yard, economy factors, etc. **Mr. Kitts.**

3. Maintenance of one man-hour laboratory control for every ten to fifty cubic yards of concrete, depending upon the conditions.

4. Routine tests of aggregates as received, for specific gravity, density, fineness modulus, grading, moisture, bulking, and absorption, in connection with silt and organic matter tests at the aggregate plant.

5. Calculation and readjustment of proportions of aggregates and water as required by changes in grading, density, specific gravity, moisture and bulking of the individual aggregates.

6. Laboratory duplication of each change of proportions to check the physical characteristics expected for that grading, cement and water content.

7. Routine slump tests and making and breaking periodical test specimens.

8. Organization and systematization of laboratory control with measuring, mixing, placing and curing, maintenance of chronological records of quality, development of research data, etc.

The quality of concrete has been maintained by this system of control on the Exchequer Dam and Yosemite Valley Railroad relocation of the Merced Irrigation District (457,000 cubic yards), on the Melones Dam of the Oakdale and South San Joaquin Irrigation Districts (90,000 cubic yards), on the Oakland-Alameda Estuary Subway (75,000 cubic yards), on the Sutherland Dam, and on other lesser projects, all in California. The "enterprising distributor of ready-mixed concrete," of whom Mr. Doe speaks, has installed this system of laboratory control and supplies "certified concrete," certifying strength, water-cement ratio, barrels of cement per cubic yard, grading, fineness modulus, weight, density, etc., as required. Unprecedented results in strength, weight, density, economy and other qualities have been obtained and maintained with uniformity.

Mr. Warren. MR. G. E. WARREN[1] (*by letter*).—The sponsors of this splendid program are to be congratulated. The changes which have recently taken place and which are now going on in concrete engineering have been furthered, as much, if not more, by the work of Committee C-9 on Concrete and Concrete Aggregates, the committee responsible for this symposium, than by any other agency. For several years the committee has been making investigations which have led to several important specifications and methods of tests. Inasmuch as these specifications are now coming into such wide use, it seems especially valuable to have such discussions as those included in the papers of

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[1] Assistant General Manager, Portland Cement Association, Chicago, Ill.

Mr. Warren. the symposium which bring to our attention so forcefully the relation of tests and specifications to field practice.

The papers by Messrs. Crum, Doe and Young have stressed the urgent need for more careful supervision of each of the factors that affects the quality of concrete. While the discussions have been confined largely to the strength of concrete the points that have been brought out are of equal importance in their relation to other qualities. So much emphasis has been placed on strength and the factors which control it as to have led some to the belief that the other factors are relatively unimportant or that information is not available regarding them.

Strength, of course, stands out as the most important requirement for most structures but often the other properties are of equal, if not greater importance than strength. In many cases, watertightness and the ability to withstand weathering are the first requisites of concrete while its strength is of secondary consideration. Fortunately, however, the factors that improve strength have a similar influence on these other qualities. With minor exceptions, it will be found that reduction in amount of mixing water, more thorough mixing and better placing and curing will improve the density and impermeability as well as the strength.

The method of handling concrete after it leaves the mixer has probably a greater influence on the durability of concrete than on its strength. Mr. Doe has very ably dealt with the methods and equipment for conveying and placing concrete which should result in improved quality. The method to be used in any case should be selected with a view to the production of a weather-resistant and durable structure.

The production of weather-resistant and durable structures involves first of all the making of a watertight concrete. To produce a watertight concrete, there are two essential requirements that must be fulfilled. First, the mixture must be so designed that it will itself resist the penetration of moisture when properly hardened, and second, it must be transported in a homogeneous condition and placed in such a way that every portion of the form is thoroughly filled with a uniform mass. The problem, therefore, is more than the mere design of a watertight mixture. It demands in addition, proper equipment, skillful handling, and competent inspection throughout the progress of the work. However, the properties of the concrete mixture have such an influence on the design of the equipment and method of handling that it must be given first consideration. The design of the mixture involves the selection of the materials and due regard to the quality included under that more or less undefinable term "workability." **Mr. Warren.**

Perhaps, in many cases where concrete structures have failed to give satisfactory results when exposed to the elements, the difficulties may be ascribed to the lack of appreciation of the importance of workability. Arbitrary mixtures have been specified, and due to the deficiencies in the materials or the proportions fixed upon, the only way the concrete could be made to puddle properly in the forms was by the addition of water. From this use of water as a necessity it is only a short transition to its more excessive use as a convenience or a transportation agency and thus has developed the practice which is now recognized to be one of the chief causes for faulty construction.

It is not the purpose of this discussion to dwell upon the design of concrete mixtures, but rather to emphasize the importance, to the life of the structure, of those operations necessary after the concrete has left the mixer. These operations are so intimately affected by the character of the mix, that in a discussion of the subject, it must be presumed that a suitable mixture has been selected. The same remark can be made in reference to the selection of the materials. While deficiencies in aggregate gradings can frequently be overcome by increasing the cement content of the mixture, this is in general an undesirable as well as an uneconomical procedure. The proper selection of the materials and design of the mixture are prerequisites to any successful construction.

After the concrete has left the mixer, the operations which require attention in all details are those of transporting, placing and curing. In transporting it is particularly important to avoid segregation. Where chutes are used the slope and the mix must be so coordinated that the materials do not segregate. Where buggies are used the runways should be constructed to give a smooth running surface free from jars and bumps. As stated by Mr. Doe, bottom dumping devices are to be preferred since some remixing action is obtained.

No operation in concrete production requires more care than the placing of the concrete in the forms. Constant supervision is essential to insure a complete filling of the form and the incorporation of the reinforcement. The practice of depositing continuously at one point, allowing the concrete to flow considerable distances, should never be permitted. In placing concrete in deep layers, a gradual increase in the water content of the upper portion results from the increased pressure on the lower portions. The effect of this increased water

Mr. Warren. content is to increase the porosity in these areas. The extra water should be absorbed by gradually reducing the quantity of water in the mix or where this is impracticable it should be removed by working it to a low point without actually causing a flow.

Perhaps no operation adds so much to the quality of concrete at so little expense as proper curing. Not only is the strength appreciably increased, but the other desirable qualities are favorably affected. Differences in the manner and extent of curing on the component parts of the same structure may often account for an apparent discrepancy in the ability to withstand the elements when other causes cannot be assigned. Withey[1] has shown the effect of curing on the permeability of concrete. He concludes that "To secure a high degree of imperviousness, concrete should be cured in a moist atmosphere or under water. At temperatures of 60 or 75° F., the moist-curing period should be maintained for two weeks to a month after molding. Lean mixes or thin sections require longer wet-curing periods than rich mixes or thick sections." Since watertight concrete is necessary in the production of weather-resistant and durable structures, too much attention cannot be given to the curing. The effect of temperature on the rate of hardening of concrete is well known, and in at least the early stages of hardening, sufficiently high temperatures should be maintained so that a large part of the ultimate strength is obtained before the forms are removed. Mr. Young brings to our attention that differences in temperature may often account for variable results in field tests, and the effect is no less in the structure itself. The relation of the design of a structure to the field operations required in its construction is a subject now being given attention, but one which will merit further study, especially with reference to proper assembly and security of reinforcing steel.

So much attention has been given to the design of concrete mixes that the interests of engineers have been more or less focused on this feature, but the papers in this symposium should be of considerable value in emphasizing the need for careful supervision of every operation if strict field control is to be the result.

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[1] M. O. Withey, "Permeability Tests on Broken Stone Concrete," Engineering Series, Vol. IX, No. 2, University of Wisconsin Engineering Experiment Station.

THE USE OF LUMNITE CEMENT IN SHORT-TIME TESTS TO DETERMINE THE QUALITY OF FINE AGGREGATE FOR CONCRETE

BY SANFORD E. THOMPSON[1] AND MILES N. CLAIR.[2]

SYNOPSIS

This paper describes a method of test, using Lumnite cement, giving the mortar strength properties of fine aggregates for concrete at 24 hours. Data are given from commercial tests made over a number of years on about two hundred fine aggregates from various parts of the country. The principal characteristics of the fine aggregates are given in most cases.

The data makes possible a comparison of this test with the results obtained from the usual portland cement mortar test at 3, 7 and 28-day periods. The results of the 24-hour mortar test using Lumnite cement compare favorably with those for the 28-day period using portland cement and is recommended for use where information as to mortar strength is required within a limited time.

INTRODUCTION

The large amount of information being made available concerning the various factors affecting the quality of concrete is making engineers realize that many items previously given but slight attention are of vital importance from the standpoint of economy and durability.

This interest in quality control is reflected in the more general use in specifications of the requirement that materials be subjected to complete laboratory examination preliminary to their use on the job. The period between the date of the signing of the contract for construction and the time the materials are required for the first concrete is often a matter of only a few weeks. If the time required to collect the samples of materials and to get them to the testing laboratory is considered, it is obvious that time available for the tests is usually a matter of a few days. Most of the present standard tests of materials entering into concrete require from 7 to 28 days before a definite rejection or acceptance can be made. The result is, generally, that either the material is used without awaiting the results of the laboratory examination or the job is held up.

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[1] President, The Thompson & Lichtner Co., Inc., Boston, Mass.

[2] Construction Engineer, The Thompson & Lichtner Co., Inc., Boston, Mass.

Various organizations have developed certain tests to enable them to identify within a very short time those materials definitely satisfactory or definitely unsatisfactory. The 1-day neat test gives a good indication when information is desired within a short time on a particular lot of cement. Likewise, a 3-day tension test of mortar briquets has been used to supplement the standard 7 and 28-day mortar test for fine aggregates.

This paper was prepared to bring to the attention of those who have to do with the testing and selection of fine aggregates for concrete, data from the use in commercial testing practice of a mortar-strength briquet test made at 24 hours using Lumnite cement. There will be given a short statement of the method of making this test, a tabulation of data for comparison of the Lumnite tests with the portland-cement mortar tests at 3, 7 and 28 days, and a brief discussion of these data.

The demand for quick information in regard to the quality of fine aggregate led the organization with which the authors are connected to experiment with and adopt the 3-day portland-cement mortar test, about six years ago, to supplement the usual 7 and 28-day tests. Preliminary experiments showed that three days was about the shortest period that could be used to give consistent results. The tests were made in accordance with the A.S.T.M. standards except that an extra set of the briquets was made up for test at three days. However, three days was too long a period in many cases, and, therefore, rather than base a preliminary rejection or acceptance on the information given by the usual examination and chemical tests alone, experimentation was started on a 24-hour test. The preliminary tests with Lumnite cement showed that it gave results that were reasonably concordant with those obtained by the standard method and so about the middle of 1925 the 3-day portland-cement test was discarded and a 1-day Lumnite-cement test adopted as standard laboratory practice to supplement the portland-cement tests at 7 and 28 days.

TEST PROCEDURE

The procedure used for this test is in accordance with the requirements of the A.S.T.M. Specifications for Concrete Aggregates (C 33 – 26 T), except that certain details which are not definitely stated in the specifications have been standardized for our work. A batch using 750 g. of standard sand, 250 g. of cement and a quantity of water based on the normal consistency of the cement, is made up and the consistency of this batch determined by a small flow table.

TABLE I.—RESULTS OF TESTS ON FINE AGGREGATE, INCLUDING BOTH LUMNITE AND PORTLAND CEMENT TESTS.

Test	Source of Fine Aggregate	Fineness Modulus	Silt, per cent	Organic Matter, parts per million of tannic acid	Strength Ratio			Remarks
					Lumnite Cement	Portland Cement		
					24 Hours	7 Days	28 Days	
No. 1	Eastern Massachusetts	2.95	0.5	none	138	145	140	Bank sand.
No. 2	Western Massachusetts	3.40	0.86	none	137	139	136	Lacking in fines. Bank sand.
No. 3	Eastern Massachusetts	3.07	2.5	20	161	167	158	Bank sand.
No. 4	Western Massachusetts	3.93	2.8	500	158	151	149	Bank sand.
No. 5	Eastern Massachusetts	3.23	1.8	none	146	137	123	Bank sand.
No. 6	Western Massachusetts	3.16	4.0	20	131	124	128	Bank sand. Excess of material passing No. 100 sieve.
No. 7	Eastern Massachusetts	3.87	2.5	100	134	140	127	Bank sand.
No. 8	Eastern Massachusetts	2.45	0.8	20	104	100	102	High in fine material. Bank sand.
No. 9	Eastern Massachusetts	2.25	2.1	100	110	105	100	Bank sand.
No. 10	Eastern Massachusetts	2.26	3.5	20	127	100	100	Bank sand.
No. 11	Eastern Massachusetts	2.72	3.0	20	115	111	115	River sand.
No. 12	Central Massachusetts	3.24	0.3	20	97	99	89	Bank sand.
No. 13	Central Massachusetts	2.18	2.6	250	87	82	83	Bank sand. Excessive fines.
No. 14	Eastern Massachusetts	3.00	2.8	none	135	154	155	Bank sand.
No. 15	Western Massachusetts	2.89	8.0	100	85	81	98	Bank sand.
No. 16	Western Massachusetts	2.25	4.4	20	96	114	117	Excess fines. Bank sand.
No. 17	Western Massachusetts	2.84	4.0	20	135	108	122	Excess fines. Bank sand.
No. 18	Western Massachusetts	3.16	5.0	20	106	111	115	Excess fines. Bank sand.
No. 19	Eastern Massachusetts	2.37	1.2	100	112	105	100	Bank sand.
No. 20	Eastern Massachusetts	....	...	20	114	108	97	Bank sand.
No. 21	Eastern Massachusetts	....	...	20	101	108	97	Bank sand.
No. 22	Eastern Massachusetts	2.44	1.0	none	118	116	120	Bank sand.
No. 23	Eastern Massachusetts	2.62	7.0	250	129	132	129	Excess fines. Bank sand.
No. 24	Eastern Massachusetts	3.23	1.0	20	131	141	119	Irregular. Bank sand.
No. 25	Central Connecticut	2.47	0.5	20	104	97	88	Poor. Bank sand.
No. 26	Central Connecticut	2.57	3.0	250	114	103	101	Bank sand.
No. 27	Maine	1.88	...	none	118	113	116	Passable. Bank sand
No. 28	Central Maine	2.40	1.5	20	100	114	113	Bank sand.
No. 29	Southeastern Maine	3.84	...	none	132	145	134	Bank sand.
No. 30	Western New York	2.86	4.9	20	124	138	136	Good. Bank sand.
No. 31	Southeastern New York	4.34	...	20	100	110	102	River sand.
No. 32	Western New York	2.72	1.1	100	87	101	103	Bank sand. Irregular.
No. 33	Eastern New York	2.64	2.2	20	81	86	81	Beach sand.
No. 34	Southern New Jersey	2.74	0.3	20	144	120	123	Bank sand.
No. 35	Southern New Jersey	2.52	0.8	100	95	108	102	Bank sand.
No. 36	Southern New Jersey	2.08	1.2	20	98	96	93	Excess fine. River sand.
No. 37	Southern New Jersey	3.04	0.2	none	144	138	116	Lacks fines. River sand.
No. 38	Southern New Jersey	2.45	0.4	250	121	107	102	River sand.
No. 39	Southern New Jersey	2.54	0.1	100	118	120	113	Bank sand.
No. 40	Southern New Jersey	2.60	0.2	100	100	117	114	River sand.
No. 41	Northeastern New Jersey	2.25	3.8	20	113	116	113	River sand.
No. 42	Central New Jersey	1.86	0.9	none	95	95	97	Beach sand.
No. 43	Northern Maryland	2.87	1.0	20	105	112	115	River sand.
No. 44	Central Tennessee	....	...	200	125	89	92	Excess fine. River sand.
No. 45	Central Tennessee	....	...	250	70	83	78	Excess fine. River sand.
No. 46	Central Tennessee	....	...	200	93	121	93	River sand.
No. 47	Central Tennessee	....	...	200	82	115	86	River sand.
No. 48	Tennessee	2.94	1.2	none	110	147	154	Bank sand.
No. 49	Tennessee	3.03	0.7	100	83	103	83	River sand.
No. 50	Tennessee	1.46	6.2	20	53	68	58	River sand.
No. 51	Tennessee	2.68	0.3	20	88	95	91	River sand.
No. 52	Tennessee	2.10	0.8	250	84	86	79	Excess fine. Bank sand.
No. 53	Tennessee	2.74	0.4	200	92	93	82	Irregular. Bank sand.
No. 54	Tennessee	2.88	0.2	20	100	100	98	River sand.
No. 55	Delaware	2.92	1.2	100	138	131	139	Bank sand.
No. 56	West Virginia	2.39	0.4	250	58	81	85	Irregular. River sand.
No. 57	West Virginia	2.37	0.5	250	115	100	106	Poor. River sand.
No. 58	West Virginia	2.60	0.5	250	100	95	99	River sand.
No. 59	South Carolina	2.98	passable	20	96	101	96	Bank sand.
No. 60	South Carolina	2.31	excess	20	74	82	72	Bank sand.
No. 61	South Carolina	2.27	excess	20	60	87	76	Passable. Bank sand.
No. 62	South Carolina	3.04	0.5	20	60	87	76	Bank sand.
No. 63	South Carolina	3.39	0.4	250	121	118	115	Bank sand.
No. 64	South Carolina	2.98	...	80	100	93	83	Bank sand.
No. 65	Louisiana	2.16	1.3	20	100	103	98	High fine. River sand.

TABLE I.—RESULTS OF TESTS ON FINE AGGREGATE, INCLUDING BOTH LUMNITE AND PORTLAND CEMENT TESTS (*Continued*).

Test	Source of Fine Aggregate	Fineness Modulus	Silt, per cent	Organic Matter, parts per million of tannic acid	Strength Ratio,			Remarks
					Lumnite Cement	Portland Cement		
					24 Hours	7 Days	28 Days	
No. 66	Eastern Georgia...........	2.64	0.2	137	137	139	136	River sand.
No. 67	Eastern Georgia...........	2.97	0.2	80	80	87	80	Poor. River sand.
No. 68	Georgia....................	2.10	1.0	100	85	91	83	River sand.
No. 69	Georgia....................	2.00	0.2	100	71	90	80	Bank sand.
No. 70	Azores.....................	1.92	1.5	none	43	72	68	Volcanic origin. Beach sand.
No. 71	Azores.....................	3.72	3.0	none	70	96	83	Volcanic origin. Beach sand.
No. 72	Azores.....................	2.95	0.1	none	156	158	130	Volcanic. Lacks fines. Beach sand.
No. 73	Azores.....................	3.27	...	none	94	101	90	Volcanic. Beach sand.
No. 74	Azores.....................	2.82	...	none	67	107	90	Volcanic. Beach sand.
No. 75	Azores.....................	3.12	...	none	74	110	88	Volcanic. Beach sand.
No. 76	Panama....................	2.80	3.4	100	97	115	100	Limestone. River sand.
No. 77	Panama....................	3.29	3.2	60	95	105	106	River sand.
No. 78	Central Ohio..............	3.52	1.4	20	145	156	157	River sand.
No. 79	Central Ohio..............	4.03	1.1	20	124	156	145	Coarse. River sand.
No. 80	Northern Michigan........	3.14	0.7	20	134	146	125	Bank sand.
No. 81	Northwestern Illinois.......	3.57	0.3	none	160	146	140	Coarse. Bank sand.
No. 82	Central Illinois............	2.75	0.5	100	100	100	103	Bank sand.
No. 83	Illinois.....................	1.60	0.8	none	48	37	51	Very fine. Carbon coated. Special.
No. 84	Arkansas.................	2.01	0.6	250	89	75	90	Poor. River sand.

The proper amount of water that will give the same consistency for the job sand as that of the standard sand is then determined by trial. The briquets are made using the quantity of water so determined. The water used for the Lumnite briquets is in the same proportion, but is based on the normal consistency of the Lumnite cement.

This consistency, as mentioned above, is determined by means of a small flow table. This table is similar in construction to the usual flow table used for concrete, except that the plate is only 10 in. in diameter and has a drop of ¼ in. The truncated cone used with it is 2 in. deep and tapers from a diameter of 2¾ in. at the top to 4 in. at the bottom. The procedure in using this apparatus is to fill the mold to one-third of its height with the mortar, then rod twenty-five times with a ¼-in. diameter rod; repeat for two-thirds full; and finally fill and rod. The top is levelled off by four strokes of the trowel and the cone removed at once. The table is then jigged for forty-five seconds at the rate of one drop every 1½ seconds. The average diameter of the base of the cone of mortar is measured with a caliper and this is fixed and used for the job sand.

The portland-cement briquets are stored in the standard manner. The Lumnite-cement specimens are covered with steel or glass plates on both sides and it is important that these be held tightly in place.

TABLE II.—RESULTS OF TESTS ON FINE AGGREGATES, INCLUDING PORTLAND CEMENT TESTS.

Test	Source of Fine Aggregate	Fineness Modulus	Silt, per cent	Organic Matter, parts per million of tannic acid	Strength Ratio, Portland Cement			Remarks
					3 Days	7 Days	28 Days	
No. 1	Massachusetts	....	...	...	121	114	108	Bank sand.
No. 2	Northeastern Massachusetts	....	...	none	101	103	102	Bank sand.
No. 3	Northeastern Massachusetts	....	...	none	102	102	101	Bank sand.
No. 4	Northeastern Massachusetts	3.58	...	none	155	121	141	Bank sand.
No. 5	Northeastern Massachusetts	2.86	...	none	124	112	102	Bank sand.
No. 6	Northeastern Massachusetts	3.06	...	none	114	102	102	Bank sand.
No. 7	Central Massachusetts	....	...	100	100	104	87	Bank sand.
No. 8	Massachusetts	....	...	500	6	4	46	Band sand.
No. 9	Massachusetts	....	...	none	100	100	95	Bank sand.
No. 10	Eastern Massachusetts	....	...	none	100	109	86	Bank sand.
No. 11	Massachusetts	....	...	none	98	102	111	Bank sand.
No. 12	Northeastern Massachusetts	....	...	none	95	100	101	Bank sand.
No. 13	Northeastern Massachusetts	....	...	none	100	161	100	Bank sand.
No. 14	Northeastern Massachusetts	....	...	none	96	101	101	Bank sand.
No. 15	Northeastern Massachusetts	....	...	none	101	102	103	Bank sand.
No. 16	Massachusetts	....	...	none	101	116	125	Bank sand.
No. 17	Massachusetts	....	...	none	117	125	110	River sand.
No. 18	Massachusetts	....	...	none	96	101	101	Bank sand.
No. 19	Massachusetts	....	...	none	105	120	107	Bank sand.
No. 20	Massachusetts	2.06	...	none	57	71	80	Bank sand.
No. 21	Massachusetts	....	...	none	119	100	100	Bank sand.
No. 22	Massachusetts	....	...	none	123	109	101	Bank sand.
No. 23	Massachusetts	....	...	20	104	103	107	Bank sand.
No. 24	Massachusetts	....	...	none	101	116	125	Bank sand.
No. 25	Massachusetts	....	...	none	101	100	102	Bank sand.
No. 26	Massachusetts	3.85	...	none	102	107	103	Bank sand.
No. 27	Massachusetts	....	...	none	113	100	101	Bank sand.
No. 28	Massachusetts	....	...	none	102	105	105	Bank sand.
No. 29	Massachusetts	....	...	350	145	143	118	Bank sand.
No. 30	Massachusetts	2.39	3.1	300	96	86	82	Bank sand.
No. 31	Massachusetts	3.13	...	none	132	129	121	River sand.
No. 32	Massachusetts	3.43	0.5	20	123	138	118	Bank sand.
No. 33	Massachusetts	2.36	..	250	125	116	93	Bank sand.
No. 34	Massachusetts	3.57	...	350	146	146	127	Bank sand.
No. 35	Massachusetts	2.50	...	none	100	100	103	Bank sand.
No. 36	Massachusetts	3.10	...	none	113	115	106	Bank sand.
No. 37	Massachusetts	2.55	...	none	101	100	100	Bank sand.
No. 38	Massachusetts	2.66	...	none	92	94	100	Bank sand.
No. 39	Massachusetts	2.67	...	none	123	112	112	Irregular. Bank sand.
No. 40	Massachusetts	3.14	...	none	114	112	112	Irregular. Bank sand.
No. 41	Massachusetts	3.10	...	500	33	86	57	Coarse. Bank sand.
No. 42	Massachusetts	2.67	...	none	100	102	102	Bank sand.
No. 43	Massachusetts	2.55	...	none	99	100	108	Bank sand.
No. 44	Massachusetts	2.73	...	none	96	125	115	Bank sand.
No. 45	Massachusetts	3.10	excess	none	113	115	106	Bank sand.
No. 46	Massachusetts	2.55	2.0	none	100	113	102	Bank sand.
No. 47	Massachusetts	3.42	5.4	none	129	141	111	Bank sand.
No. 48	Massachusetts	2.44	0.76	none	106	121	121	Bank sand.
No. 49	Massachusetts	3.10	0.64	none	121	142	148	Bank sand.
No. 50	Massachusetts	2.76	...	100	104	131	112	Bank sand.
No. 51	Massachusetts	2.12	...	100	74	81	80	Excess fines. Bank sand.
No. 52	Massachusetts	2.21	2	20	126	122	93	Bank sand.
No. 53	Western Massachusetts	....	...	none	150	136	118	Bank sand.
No. 54	Massachusetts	....	...	none	123	124	140	Bank sand.
No. 55	Western Massachusetts	....	...	none	74	80	85	Bank sand.
No. 56	Massachusetts	....	...	none	92	95	101	Bank sand
No. 57	Massachusetts	2.75	...	none	112	119	115	Bank sand.
No. 58	Massachusetts	....	...	none	114	20	109	Bank sand.
No. 59	Massachusetts	2.35	...	none	128	128	120	Bank sand.
No. 60	Massachusetts	....	...	350	71	92	90	Bank sand.
No. 61	Massachusetts	....	...	none	136	146	125	Bank sand.
No. 62	Massachusetts	....	...	none	131	127	113	Bank sand.
No. 63	Massachusetts	3.01	...	none	131	107	105	Bank sand.
No. 64	Massachusetts	....	...	none	114	120	103	Bank sand.
No. 65	Massachusetts	2.26	...	none	134	125	104	Bank sand.
No. 66	Maine	3.33	...	none	103	105	104	Bank sand.
No. 67	Maine	2.24	...	none	103	105	104	Bank sand.
No. 68	Maine	....	...	100	82	93	92	Bank sand.
No. 69	Maine	....	...	350	72	72	79	Bank sand.
No. 70	Southeastern Maine	....	...	none	78	100	100	Bank sand.

TABLE II.—RESULTS OF TESTS ON FINE AGGREGATES, INCLUDING PORTLAND CEMENT TESTS (*Continued*).

Test	Source of Fine Aggregate	Fineness Modulus	Silt, per cent	Organic Matter, parts per million of tannic acid	Strength Ratio Portland Cement			Remarks
					3 Days	7 Days	28 Days	
No. 71	Southeastern Maine	....	...	none	95	89	91	Bank sand.
No. 72	Southeastern Maine	....	...	none	92	77	82	Bank sand.
No. 73	Southeastern Maine	....	...	none	120	141	136	Bank sand.
No. 74	Maine	2.45	...	none	82	106	109	Bank sand.
No. 75	Maine	2.52	...	none	83	109	109	Irregular. Bank sand.
No. 76	Maine	2.61	...	20	53	93	91	Irregular. Bank sand.
No. 77	Maine	2.38	...	none	101	126	129	Bank sand.
No 78	South Carolina	2.03	...	none	102	102	101	Bank sand.
No. 79	Vermont	....	...	100	110	102	101	Bank sand.
No. 80	Vermont	....	...	500	46	31	23	Bank sand.
No. 81	Connecticut	....	...	5	30	56	67	Bank sand.
No. 82	Connecticut	....	...	100	89	96	98	Bank sand.
No. 83	Connecticut	....	...	100	85	96	100	Bank sand.
No. 84	Connecticut	....	...	none	110	119	123	Bank sand.
No. 85	Connecticut	2.31	...	none	100	111	103	Excess fines. Bank sand.
No. 86	New Hampshire	....	...	none	111	104	102	Bank sand.
No. 87	New Hampshire	....	...	350	78	94	85	Bank sand.
No. 88	New Hampshire	2.02	...	none	131	107	105	Bank sand.
No. 89	New Hampshire	1.77	...	none	112	119	115	Bank sand.
No. 90	New York	....	...	none	102	102	101	Bank sand.
No. 91	Eastern Pennsylvania	....	...	none	131	131	152	Slag.
No. 92	Central Pennsylvania	2.81	...	none	108	115	118	Bank sand.
No. 93	Central Pennsylvania	1.66	...	350	62	63	75	Bank sand.
No. 94	Pennsylvania	....	...	none	106	102	103	Bank sand.
No. 95	Pennsylvania	....	...	none	71	65	98	Bank sand.
No. 96	Eastern Pennsylvania	....	...	none	84	84	95	Slag.
No. 97	New Jersey	....	...	none	106	112	110	Bank sand.
No. 98	Maryland	....	...	none	132	129	113	Bank sand.
No. 99	District of Columbia	....	...	none	116	101	99	River sand.
No. 100	District of Columbia	....	...	none	75	68	88	River sand.
No 101	Tennessee	....	...	20	96	93	98	Bank sand.
No. 102	Tennessee	....	...	20	100	102	105	Bank sand.
No. 103	Tennessee	2.58	...	none	88	91	92	Bank sand.
No. 104	Tennessee	2.93	...	20	87	98	89	Irregular. Bank sand.
No. 105	Tennessee	2.83	...	250	102	102	101	Irregular. Bank sand.
No. 106	Kentucky	3.40	...	none	161	151	148	Bank sand.
No. 107	Kentucky	3.18	...	100	59	73	85	Bank sand.
No. 108	North Carolina	2.99	0.2	20	100	103	107	Bank sand.
No. 109	Florida	....	...	none	100	100	101	Excess fines. River sand.
No. 110	New York	....	...	none	95	101	136	Bank sand.
No. 111	Minnesota	3.27	...	none	145	139	121	Soft stone. Bank sand.
No. 112	Minnesota	....	...	none	104	131	100	Bank sand.

The essential requirements for success in the use of this method is the absolute prevention of contamination of the Lumnite cement by portland cement, lime and calcium chloride, and use of the plates on both sides of the specimens. It is preferable that entirely separate tables be used for the two cements. Failure to observe these precautions will result in faulty briquets.

TEST DATA

Table I gives data from commercial tests made on fine aggregate for concrete using the Lumnite-cement 24-hour test to supplement the usual 7 and 28-day portland-cement tests. The data are not selected data, but represent all that it is feasible to present at this time. The tests were made by several different operators using seven different brands of portland cement. Certain characteristics of the materials are indicated where available. The strength ratio for the Lumnite cement is based on Lumnite-cement briquets as 100 made at the same time using standard Ottawa sand. The strength ratio for the portland cement is based on portland-cement briquets as 100 made at the same time as the Lumnite briquets and using standard sand. Under the heading "Remarks" is included information as to the kind of sand and its grading. Sand concerning which there are no remarks may in general be considered as being of fairly satisfactory grading.

Table II is given for comparison of the 3-day mortar test with the Lumnite-cement tests given in Table I. It presents data from commercial tests of fine aggregate for concrete made during a period of three to four years prior to the Lumnite tests. There was much less information available in regard to the characteristics of those materials than for those tested during the last two years. The strength ratio for the 3-day portland-cement briquets is based on a corresponding set using standard sand as 100 and made at the same time.

DISCUSSION

The data in Table I show considerable variation in individual cases between the Lumnite-cement test and the portland-cement 28-day test which is taken as standard. The average mean difference, however, does not exceed 10 per cent which compares favorably with 8 per cent for the 7-day test. The Lumnite test tends to be but slightly optimistic while the 7-day test is decidedly so.

We use a mortar-strength ratio of 85 per cent at 28 days as the lowest acceptable for a concrete aggregate which shows a high organic content. Using this as a basis, it would appear from the table that only four would have been rejected which were usable and two would have been accepted which were not usable. Actually, other factors, such as organic matter, governed in all of these cases so that in no case did the Lumnite test cause an error in selection of aggregate.

Comparing the information of Table II with that of Table I, it is seen that the two methods of test show about the same average variation from the 28-day ratio, but the 3-day test is slightly pessimistic while the Lumnite test is slightly optimistic.

The correlation coefficient with their probable errors for the several cases are as follows:

1-day Lumnite and 28-day portland	0.867 ± 0.018
3-day portland and 28-day portland	0.714 ± 0.031
7-day portland and 28-day portland	0.860 ± 0.013

These coefficients show (a correlative coefficient of 1.000 is perfect) that there is a slightly higher degree of relationship for the 1-day Lumnite and the 28-day portland test than for the 7-day and 28-day portland test.

CONCLUSION

The Lumnite-cement 24-hour mortar test for determining the quality of fine aggregate for concrete, made in accordance with the method described in this paper, gives results comparable with those obtained by the portland-cement mortar test at 28 days. We consider it of value where information must be obtained concerning the qualities of fine aggregate within 24 hours and which is to be checked by portland-cement tests later.

DISCUSSION

Mr. Smith.

Mr. W. Furber Smith[1] (*by letter*).—The method described for and results obtained by the use of Lumnite cement in short-time tests are by far the best that have come to the writer's attention to date. In spite of this fact, a rather serious objection occurs to one who is working under specifications demanding a minimum strength ratio of 100 per cent at 28 days. A glance through the data shown in Table I of the paper indicates that, had the higher strength ratio been the criterion, five erroneous rejections and eight erroneous acceptances would have been made out of 84 samples—15 per cent of erroneous decisions in all. While this percentage of error is lower than that obtained in a similar but rather brief study of the subject in this laboratory it is still high enough to seriously reduce the merit of the method for much state and commercial work.

Messrs. Thompson and Clair.

Messrs. Sanford E. Thompson[2] and Miles N. Clair[3] (*authors' closure by letter*).—If the testing of materials were a matter of absolute accuracy, the objection mentioned by Mr. Smith would tend to reduce the value of the lumnite test.

The average deficiency of the lumnite ratio below the 28-day standard for erroneous rejections on the basis of the 100-per-cent rate is 12 per cent, and the average excess for erroneous acceptance is also 12 per cent. These values are certainly not unusual, considering the 15-per-cent variation between breaks allowable in the considerably better controlled standard cement-mortar test.

It must be recognized, of course, that the 24-hour test is not necessarily final, but like the 7-day with portland cement, may be subject to 28-day results with portland cement. In practice we have found it extremely useful in giving an advance opinion in the quality of the aggregates.

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[1] Assistant Testing Engineer, Oklahoma State Highway Commission, Oklahoma City, Okla.

[2] President, The Thompson & Lichtner Co., Inc., Boston, Mass.

[3] Construction Engineer, The Thompson & Lichtner Co., Inc., Boston, Mass.

IMPROVED BRICK MORTARS

By Laurence E. Weymouth[1]

Synopsis

This paper presents the results of a series of tests on sixty different brick mortars, ranging from very plastic to very lean mixes. The mixes were made up to the same consistency as measured by the slump of a 2 by 2½-in. cylinder of the mortar. The plasticity was measured on an Emley plasticimeter according to a method modified somewhat from the standard A.S.T.M. method for the plasticity of hydrated lime. Six 3 by 6-in. cylinders and six standard briquets were made in each case for compressive and tensile strengths at 28 days and 6 months. The specimens were stored in a damp room. Five pairs of brick were laid up with each mix and tested at 28 days to measure the adhesive strength of the bond. The times of initial and final set and the weight per cubic foot were observed in each case. Three cement-sand ratios were used, namely, 1:6, 1:12 and 1:18 by volume, and varying proportions of hydrated lime and diatomaceous silica. Considerable time was spent in working out satisfactory methods of testing, as there are at present practically no standard tests applicable to brick mortars.

The results bring out some interesting relations between the plasticity, strength and composition of mortar mixes and show particularly how a large increase in strength may be obtained by the use of diatomaceous silica.

Introduction

There are very few published data available upon the properties of brick mortars. This is largely due to the difficulty of measuring the very important property of plasticity and to the lack of any standardization of tests for other properties. It is hoped that the data here presented, covering tests on a fairly comprehensive series of sixty mortars ranging from very plastic to very lean mixes, will stimulate interest in this important and neglected field.

Two important properties of a brick mortar are plasticity and strength. The strength includes that of the bond between the mortar and brick as well as the compressive and tensile strength of the mortar itself. Building codes often include strength specifications but there is much indefiniteness as to what properties are most desirable in a mortar. Increased strength is undoubtedly desirable, especially when it can be obtained without undue sacrifice of economy and of the desired plastic qualities.

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[1] Chemical Engineer, Celite Products Co., Lompoc, Calif.

If it is possible to increase the strength of mortars by changes in mixtures, it will be possible not only to secure such increased strengths where it is advisable but, on the other hand, to design more economical mixes which will give equal safety and at the same time with no sacrifice in plasticity or workability. This possibility of more economical mortars is of particular interest in the case of cities which now specify very rich mortar mixes which could in some cases be changed to advantage.

TABLE I.—BRICK MORTAR MIXES BY VOLUME.

Proportions given in order of Hydrated Lime : Cement : Sand : Diatomaceous Silica.

Lime-Sand Ratio	Mix	A	B	C	D
		Hydrated Lime-Cement-Sand Mortar	25 Per Cent of Hydrated Lime in "A" Replaced by One-half Its Weight of Diatomaceous Silica	50 Per Cent of Hydrated Lime in "A" Replaced by One-half Its Weight of Diatomaceous Silica	75 Per Cent of Hydrated Lime in "A" Replaced by One-half Its Weight of Diatomaceous Silica
1:2	No. 1	$1\frac{1}{2}:\frac{1}{6}:3$	$1\frac{1}{8}:\frac{1}{6}:3:\frac{3}{4}$	$\frac{3}{4}:\frac{1}{6}:3:1\frac{1}{2}$	$\frac{3}{8}:\frac{1}{6}:3:2\frac{1}{4}$
	No. 2	$1\frac{1}{2}:\frac{1}{3}:3$	$1\frac{1}{8}:\frac{1}{3}:3:\frac{3}{4}$	$\frac{3}{4}:\frac{1}{3}:3:1\frac{1}{2}$	$\frac{3}{8}:\frac{1}{3}:3:2\frac{1}{4}$
	No. 3	$1\frac{1}{2}:\frac{1}{2}:3$	$1\frac{1}{8}:\frac{1}{2}:3:\frac{3}{4}$	$\frac{3}{4}:\frac{1}{2}:3:1\frac{1}{2}$	$\frac{3}{8}:\frac{1}{2}:3:2\frac{1}{4}$
1:2.4	No. 4	$1\frac{1}{4}:\frac{1}{6}:3$	$\frac{15}{16}:\frac{1}{6}:3:\frac{5}{8}$	$\frac{5}{8}:\frac{1}{6}:3:1\frac{1}{4}$	$\frac{5}{16}:\frac{1}{6}:3:1\frac{7}{8}$
	No. 5	$1\frac{1}{4}:\frac{1}{3}:3$	$\frac{15}{16}:\frac{1}{3}:3:\frac{5}{8}$	$\frac{5}{8}:\frac{1}{3}:3:1\frac{1}{4}$	$\frac{5}{16}:\frac{1}{3}:3:1\frac{7}{8}$
	No. 6	$1\frac{1}{4}:\frac{1}{2}:3$	$\frac{15}{16}:\frac{1}{2}:3:\frac{5}{8}$	$\frac{5}{8}:\frac{1}{2}:3:1\frac{1}{4}$	$\frac{5}{16}:\frac{1}{2}:3:1\frac{7}{8}$
1:3	No. 7	$1:\frac{1}{6}:3$	$\frac{3}{4}:\frac{1}{6}:3:\frac{1}{2}$	$\frac{1}{2}:\frac{1}{6}:3:1$	$\frac{1}{4}:\frac{1}{6}:3:1\frac{1}{2}$
	No. 8	$1:\frac{1}{3}:3$	$\frac{3}{4}:\frac{1}{3}:3:\frac{1}{2}$	$\frac{1}{2}:\frac{1}{3}:3:1$	$\frac{1}{4}:\frac{1}{3}:3:1\frac{1}{2}$
	No. 9	$1:\frac{1}{2}:3$	$\frac{3}{4}:\frac{1}{2}:3:\frac{1}{2}$	$\frac{1}{2}:\frac{1}{2}:3:1$	$\frac{1}{4}:\frac{1}{2}:3:1\frac{1}{2}$
1:4	No. 10	$\frac{3}{4}:\frac{1}{6}:3$	$\frac{9}{16}:\frac{1}{6}:3:\frac{3}{8}$	$\frac{3}{8}:\frac{1}{6}:3:\frac{3}{4}$	$\frac{3}{16}:\frac{1}{6}:3:1\frac{1}{8}$
	No. 11	$\frac{3}{4}:\frac{1}{3}:3$	$\frac{9}{16}:\frac{1}{3}:3:\frac{3}{8}$	$\frac{3}{8}:\frac{1}{3}:3:\frac{3}{4}$	$\frac{3}{16}:\frac{1}{3}:3:1\frac{1}{8}$
	No. 12	$\frac{3}{4}:\frac{1}{2}:3$	$\frac{9}{16}:\frac{1}{2}:3:\frac{3}{8}$	$\frac{3}{8}:\frac{1}{2}:3:\frac{3}{4}$	$\frac{3}{16}:\frac{1}{2}:3:1\frac{1}{8}$
1:6	No. 13	$\frac{1}{2}:\frac{1}{6}:3$	$\frac{3}{8}:\frac{1}{6}:3:\frac{1}{4}$	$\frac{1}{4}:\frac{1}{6}:3:\frac{1}{2}$	$\frac{1}{8}:\frac{1}{6}:3:\frac{3}{4}$
	No. 14	$\frac{1}{2}:\frac{1}{3}:3$	$\frac{3}{8}:\frac{1}{3}:3:\frac{1}{4}$	$\frac{1}{4}:\frac{1}{3}:3:\frac{1}{2}$	$\frac{1}{8}:\frac{1}{3}:3:\frac{3}{4}$
	No. 15	$\frac{1}{2}:\frac{1}{2}:3$	$\frac{3}{8}:\frac{1}{2}:3:\frac{1}{4}$	$\frac{1}{4}:\frac{1}{2}:3:\frac{1}{2}$	$\frac{1}{8}:\frac{1}{2}:3:\frac{3}{4}$

NOTE: Proportions based on 40 lb. per cu. ft. for hydrated lime, 94 lb. per cu. ft. for cement, 100.9 lb. per cu. ft. for sand and 10 lb. per cu. ft. for diatomaceous silica.

It is the aim of this paper to show what strengths and plasticities may be obtained with various lime-cement mortars, and particularly to show how a large increase in strength may be obtained by taking advantage of the chemical reaction between lime and diatomaceous silica.

OUTLINE OF TESTS

The tests consisted in making up and testing sixty different mortar mixes. The mixes used and the plan of tests is shown in Table I. Column A consists of five groups of straight hydrated lime-cement-sand mortars met in actual practice. In these five groups the lime-sand ratio varies from the very plastic 1:2 ratio to the lean 1:6 ratio in the lowest group. Specifically, these five basic groups contain lime-sand ratios of 1:2, 1:2.4, 1:3, 1:4 and 1:6. Each of these groups is composed of three mortars in which three proportions of cement are employed.

In columns B, C and D, 25, 50 and 75 per cent, respectively, of the hydrated lime present in the groups of column A has been replaced by half its weight (or twice its volume) of pulverulent diatomaceous silica. These proportional replacements were used inasmuch as preliminary tests had shown that such a method of replacement gave mixes of approximately equal plasticity and cost.

The mixes were all made up to the same consistency as determined by the slump of a 2 by 2½-in. cylinder and all of the tests on each mix were made on the same batch. An argument may be advanced in favor of using a drier consistency for the tensile and compressive strength test specimens than for the other tests. In laying up brick, water is absorbed from the mortar, making it, under proper conditions, a much drier and stronger mix than in the case where the water remains in the mortar. However, it was considered a fairer comparison, on the whole, to make all of the tests on the same batch of material even though this gives compressive and tensile strengths that are too low.

After bringing to the proper consistency, two plasticity tests were made on each batch, five pairs of brick were laid up for adhesion tests, a determination of weight per cubic foot was made, a time of set specimen was molded in a rubber ring, and finally six briquets and six 3 by 6-in. cylinders were molded.

Materials and Method of Making Tests

Materials.—The hydrated lime used was from a fresh shipment of high-grade commercial high-calcium product. This material conformed to the A.S.T.M. specifications for Mason's hydrated lime as to fineness, carbon dioxide content, and calcium and magnesium oxide content. The tests are as follows:

Fineness on No. 30 sieve, per cent	0.35
Fineness on No. 200 sieve, per cent	8.63
Volatile Matter, per cent	24.1
CaO in Non-Volatile Matter, per cent	95.8
MgO " " " " "	1.4
CO_2 " " " " "	1.9

The hydrated lime was mixed by emptying the sacks upon the floor of the laboratory, mixing thoroughly with a shovel, and replacing in paper sacks which were then sealed.

A commercial brand of portland cement was used which had been stored in the laboratory for approximately six months before using. An amount sufficient for this series of tests was thoroughly mixed together and resacked. The lumps were broken up before using by putting through a No. 16 sieve.

The sand used was high-grade river bottom sand from the neighborhood of Los Angeles. Material coarser than a No. 10 sieve was removed by screening. An amount sufficient for the whole series was divided into small uniform portions by repeated division into two parts through a riffle. The tests of the sand are as follows:

Weight, lb. per cu. ft.................................... 100.9 lb.

Fineness Modulus:

SIEVE	RETAINED, PER CENT
No. 4	0
No. 8	0
No. 14	15.1
No. 28	44.3
No. 48	80.6
No. 100	96.0
	236.0 ÷ 100 = 2.36 F. M.

The diatomaceous silica used was a commercial sample of "Celite for Concrete" from the Celite Products Co. It met the following requirements:

Less than 10 per cent retained on a No. 200 sieve;
More than 90 per cent SiO_2 (ignited basis);
Less than 4 per cent Al_2O_3 (ignited basis);
Less than 8 per cent moisture.

Mixing and Consistency of Mortar.—Although based on proportions by volume, each batch of material was weighed out. The hydrated lime was soaked overnight, in each case, with an equal weight of water. The cement, sand, and diatomaceous silica were first mixed dry, the hydrated lime paste added, then sufficient water was gradually added until the desired consistency was obtained. A hollow metal cylinder 2 in. in inside diameter by 2½ in. high was used for measuring the consistency. This was filled with the wet mortar, the top struck off level, the cylinder carefully raised, and the amount of slump from the original height of 2½ in. was measured at the center of the specimen, which was considerably lower than the edges. The mixes were all adjusted to a ½-in. slump.

Plasticity Tests.—The plasticity of each mix was measured immediately after mixing, in each case, by running duplicate tests on the

Emley plasticimeter according to a method modified somewhat from the standard A.S.T.M. method for the plasticity of hydrated lime.[1] The method consists in measuring the torque exerted by a specimen of mortar, molded on a porous absorbent base plate, when slowly rotated and pressed up against a disk which is free to turn. The torque exerted on this disk is measured by means of a pendulum which is swung toward a horizontal position. The absorption of the base plate gradually removes water from the specimen during the test. (See Fig. 1.)

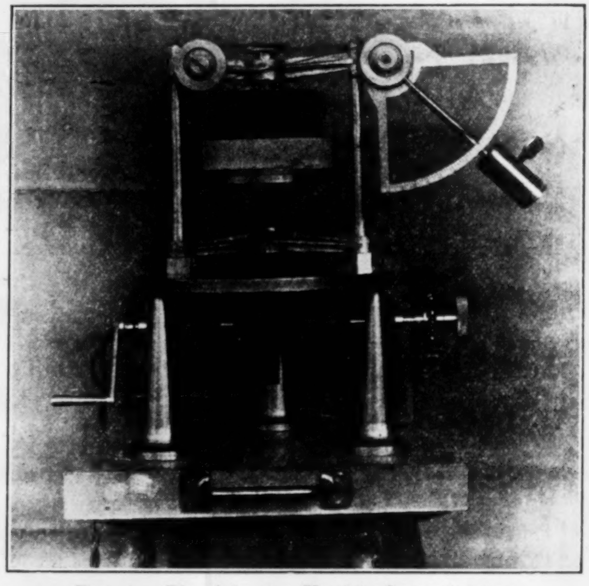

FIG. 1.—Plasticimeter Used in Investigation.

A brass ring, 3 in. in inside diameter and $1\frac{1}{2}$ in. deep was placed on the porous base plate and filled with the wet mortar. The top was struck off level. The time of placing the mortar in the mold was noted and taken as the start of the test. At exactly 1 minute the mold was raised and the specimen on the base plate placed in position on the rotating carriage. The carriage was then raised until the upper surface of the specimen was in contact with the disk and the upper surface of the base plate was exactly $1\frac{3}{8}$ in. from the disk. The clutch was then engaged. This operation was timed so that the carriage was brought into position just before the two-minute period had expired. At exactly two minutes the motor was started, slowly rotating the carriage. The position of the pendulum bob on the scale was then noted each 30 seconds until the end of the test.

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[1] See the Standard Specifications for Hydrated Lime for Structural Purposes (Serial Designation: C 6), 1927 Book of A.S.T.M. Standards, Part II.

In testing the more plastic mixes, the scale reading reached 100 without rupturing the specimen. The time at which the 100 reading was reached was noted and considered as the end of the test. In the leaner mixes the specimen ruptured before the scale reading reached 100. The time of rupture was noted in these cases as the end of the test, and the corresponding scale reading also recorded. The specimen was considered to have ruptured when a crack caused by the rupturing of the specimen extended half way down the specimen.

The plasticity figure was obtained from this data by the formula:

$$P = \sqrt{F^2 + (10T)^2}$$

in which F = the scale reading at the end of the test and T = the time in minutes from placing the mortar in the mold on the porous base plate to the end of the test. The more plastic the mortar, the higher will be this figure. The plasticity figures shown in the paper represent the average of two tests in most cases.

Adhesion Specimens.—In order to determine the adhesive force between the mortar and brick, five pairs of common building brick were laid together with each mix. The brick were laid crosswise so that they could be readily tested by pulling apart. The area of contact was, therefore, 16 sq. in. The brick were soaked for 10 seconds in water before laying up. An amount of mortar sufficient to make a joint approximately ½ in. in thickness was spread on the bottom brick, a groove made through the center with a trowel, the upper brick pressed down into position, and the edges smoothed with a trowel. The five pairs of brick were piled one above the other in the damp room.

Time of Set.—The time of initial and final set was determined in accordance with the standard A.S.T.M. method,[1] using the Vicat apparatus. This method involves keeping the specimens in a closed space over water; therefore, allowing no drying out of the specimens and very little carbonation of the lime. It was impossible to determine the time of set with absolute accuracy.

Weight per Cubic Foot.—The weight per cubic foot was determined by obtaining the weight of mortar required to fill a $\frac{1}{10}$-cu.-ft. measure. The mold was filled one-third full and tamped twenty-five times with a flat-ended steel rod ¾ by 8 in. The mold was then filled two-thirds and heaping full, tamping in the same way each time. The top was then struck off level and the full measure weighed.

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[1] See the Standard Specifications and Tests for Portland Cement (Serial Designation: C 9), 1927 Book of **A.S.T.M. Standards**, Part II.

The mortar was then emptied out, remixed, and replaced in the measure in the same manner as before. The density of the mortar usually increased each time the measure was filled, due to the compacting effect of the handling. In order to obtain as reliable values as possible, these determinations were repeated until an agreement within 0.02 lb. was reached between check tests. An average of the two values checking to 0.02 lb. or closer was taken as the weight per cubic foot of the mortar.

Compressive and Tensile Strength Specimens.—Six specimens were made for both tensile and compressive strength tests.

The tensile strength specimens were made in the form of regular cement mortar briquets in three-gang brass molds. The molds were oiled and placed on an oiled-glass plate. The molds were first approximately half filled with mortar and each specimen tamped ten times with a $\frac{3}{4}$ by 8 in. flat-ended steel rod. The molds were then completely filled and again tamped ten times, then struck off level. The specimens were removed from the molds and placed in the damp room on the next day after they were made.

The compressive strength specimens were molded in 3 by 6-in. brass cylinders set on a cast-iron base plate. The molds were filled at first approximately one-third full, and tamped ten times with a $\frac{3}{4}$ by 8 in. flat-ended steel rod. The molds were then filled two-thirds and completely full and each layer tamped in the same way. The tops were then trowelled off smooth. After a suitable time, depending on the amount of cement in the mix, the specimens were capped with neat cement paste which had previously been mixed and allowed to stand for several hours. An oiled-glass plate was pressed down upon the mold, thus making a true plane surface. The glass plate was removed when the specimen had sufficiently hardened and the specimen removed from the mold and placed in the damp room.

Storage.—The specimens were stored in a damp room in which a relative humidity of about 85 per cent was maintained.

Strength Tests.—The briquets were tested in a Fairbanks briquet machine. The compression test cylinders were tested in a Riehlé 200,000-lb. universal testing machine, using a breaking speed of 0.05 in. per minute.

The adhesive strength tests were made with a special device designed to distribute the load uniformly in breaking the specimens. The upper brick of the pair was supported between two upright planks spaced about 6 in. apart and resting on the platform of a platform scale. Loops of wire cable were passed around each end of the lower brick. A pulley was fastened at the bottom of each of

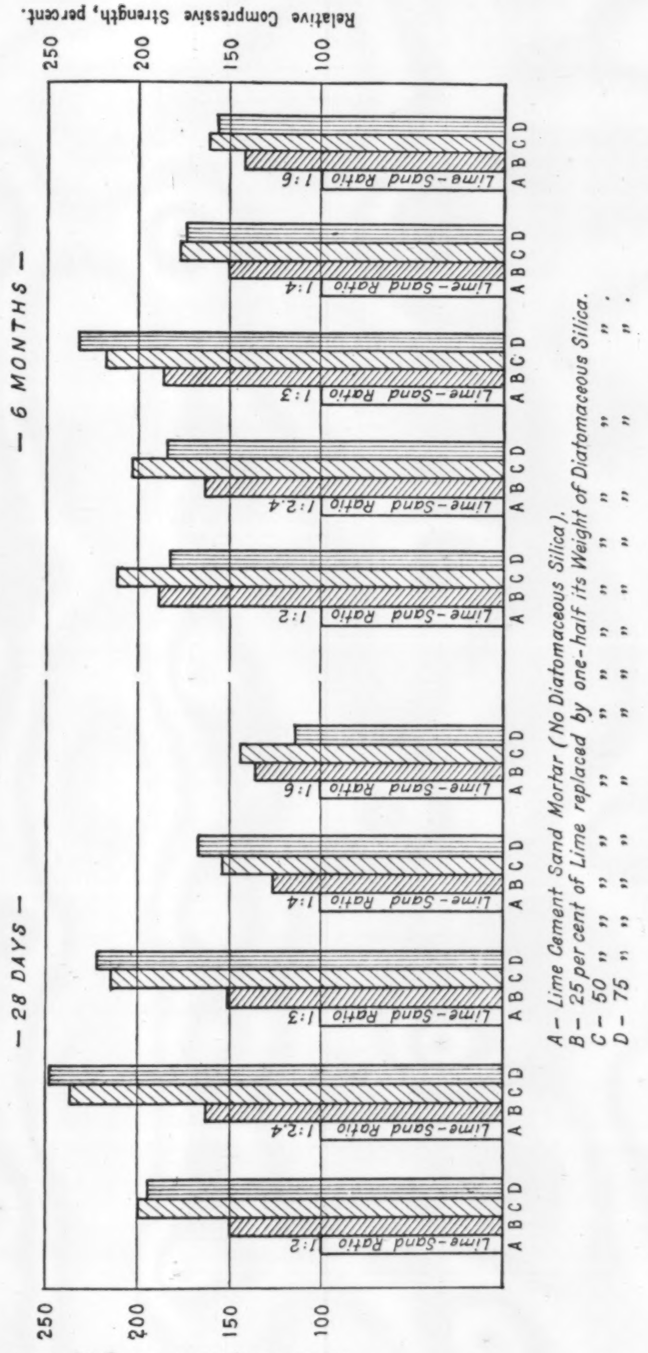

FIG. 2.—Relative Compressive Strength of Basic Groups.

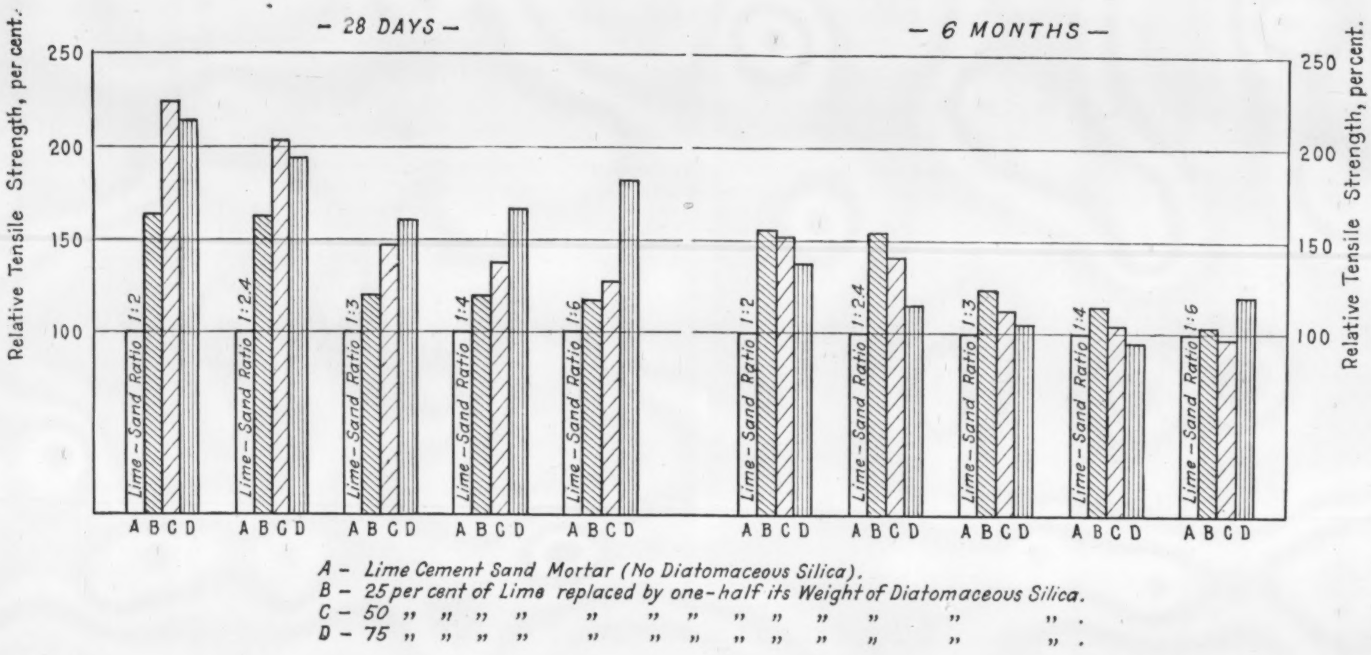

FIG. 3.—Relative Tensile Strength of Basic Groups.

these loops of cable and these two pulleys were connected by another length of cable carrying a third pulley, to which the load was applied by means of a pulley and windlass arrangement. The scales were kept in balance while the load was gradually increased, and the reading at the break noted.

DISCUSSION OF RESULTS

The results of the tests are presented in Tables II to XII, inclusive. Certain of the data are presented graphically in Figs. 2, 3 and 4.

The most striking results of this series of tests are the large increases in strength obtained by the use of diatomaceous silica. In comparison with mixes A (which contained no diatomaceous silica) the averages of all mixes tested show the following results:

AVERAGE RELATIVE STRENGTHS OF ALL MIXES AT 28 DAYS

	MIX A	MIX B	MIX C	MIX D
Compressive Strength, per cent...	100	149	190	189
Tensile Strength, per cent........	100	136	167	183

AVERAGE RELATIVE STRENGTHS OF ALL MIXES AT 6 MONTHS

	MIX A	MIX B	MIX C	MIX D
Compressive Strength, per cent...	100	167	194	187
Tensile Strength, per cent........	100	130	121	114

Furthermore, if the lean 1:6 lime-sand group (mix Nos. 13–15, Table I) which is probably not sufficiently plastic for most purposes, is left out of the average, the increased strength with diatomaceous silica is even more striking.

Tables II to VI record the strength, plasticity, and cost figures obtained for the various basic groups and replacement mixes. The relative compressive strengths of these replacement mixes, with reference to the basic mix containing no diatomaceous silica, are given graphically in Fig. 2. The relative tensile strengths for the same groups are given in Fig. 3. These groups and the results shown in the graphs embrace mixes with the three proportions of cement.

It will be noted that Table VII, composed of mixes recorded elsewhere, shows the effect of adding diatomaceous silica to a lime-cement mortar (not replacing part of the lime by diatomaceous silica). This addition greatly increases the plasticity but does not produce such a marked increase in strength as is obtained by partial replacement of the lime by diatomaceous silica according to the plan here followed, namely, that of obtaining mixes of equal plasticity and cost. The addition of diatomaceous silica is seen to produce a much greater relative increase in strength in the weaker mixes containing smaller proportions of cement than in the richer 1:6 cement-sand ratio group

TABLE II.—STRENGTH, PLASTICITY AND COST FIGURES; BASIC 1:2 LIME-SAND RATIO GROUP.

Each result is average of three determinations

	Mix by Volume[a]	Plasticity Figure	Compressive Strength				Tensile Strength				Cost per Cubic Yard of Wet Mortar, dollars
			28 Days		6 Months		28 Days		6 Months		
			lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	
No. A1	$1\frac{1}{2}:\frac{1}{6}:3:0$	130	66	100	168	100	28	100	65	100	6.87
No. B1	$1\frac{1}{8}:\frac{1}{6}:3:\frac{3}{4}$	131	99	150	421	251	46	164	87	134	6.81
No. C1	$\frac{3}{4}:\frac{1}{6}:3:1\frac{1}{2}$	111	157	238	461	275	72	257	103	158	6.87
No. D1	$\frac{3}{8}:\frac{1}{6}:3:2\frac{1}{4}$	105	145	220	330	197	60	214	94	145	6.84
No. A2	$1\frac{1}{2}:\frac{1}{3}:3:0$	129	91	100	306	100	40	100	75	100	7.24
No. B2	$1\frac{1}{8}:\frac{1}{3}:3:\frac{3}{4}$	127	147	162	555	181	65	163	152	203	7.31
No. C2	$\frac{3}{4}:\frac{1}{3}:3:1\frac{1}{2}$	130	184	202	635	208	84	210	133	177	7.32
No. D2	$\frac{3}{8}:\frac{1}{3}:3:2\frac{1}{4}$	124	191	210	661	216	87	218	122	163	7.20
No. A3	$1\frac{1}{2}:\frac{1}{2}:3:0$	129	202	100	689	100	57	100	154	100	7.78
No. B3	$1\frac{1}{8}:\frac{1}{2}:3:\frac{3}{4}$	132	280	139	938	136	93	163	199	129	7.85
No. C3	$\frac{3}{4}:\frac{1}{2}:3:1\frac{1}{2}$	127	321	159	1024	149	117	205	180	117	7.85
No. D3	$\frac{3}{8}:\frac{1}{2}:3:2\frac{1}{4}$	126	307	152	936	136	120	211	156	101	7.73

[a] Proportions given in order of Hydrated Lime : Cement : Sand : Diatomaceous Silica.

TABLE III.—STRENGTH, PLASTICITY AND COST FIGURES; BASIC 1:2.4 LIME-SAND RATIO GROUP.

Each result is average of three determinations.

	Mix by Volume[a]	Plasticity Figure	Compressive Strength				Tensile Strength				Cost per Cubic Yard of Wet Mortar, dollars
			28 Days		6 Months		28 Days		6 Months		
			lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	
No. A4	$1\frac{1}{4}:\frac{1}{6}:3:0$	103	35	100	164	100	29	100	58	100	6.33
No. B4	$\frac{15}{16}:\frac{1}{6}:3:\frac{5}{8}$	126	94	268	294	179	54	186	115	198	6.40
No. C4	$\frac{5}{8}:\frac{1}{6}:3:1\frac{1}{4}$	92	126	360	407	248	66	228	97	167	6.42
No. D4	$\frac{5}{16}:\frac{1}{6}:3:1\frac{7}{8}$	88	150	429	381	232	60	207	72	124	6.44
No. A5	$1\frac{1}{4}:\frac{1}{3}:3:0$	124	96	100	313	100	34	100	85	100	7.04
No. B5	$\frac{15}{16}:\frac{1}{3}:3:\frac{5}{8}$	118	150	156	536	171	60	177	111	131	6.95
No. C5	$\frac{5}{8}:\frac{1}{3}:3:1\frac{1}{4}$	110	197	205	675	216	76	224	113	133	6.94
No. D5	$\frac{5}{16}:\frac{1}{3}:3:1\frac{7}{8}$	109	186	194	621	198	66	194	92	108	6.85
No. A6	$1\frac{1}{4}:\frac{1}{2}:3:0$	122	261	100	698	100	74	100	154	100	7.53
No. B6	$\frac{15}{16}:\frac{1}{2}:3:\frac{5}{8}$	128	300	115	984	141	90	122	198	129	7.55
No C6	$\frac{5}{8}:\frac{1}{2}:3:1\frac{1}{4}$	131	384	147	1018	146	116	157	185	120	7.51
No. D6	$\frac{5}{16}:\frac{1}{2}:3:1\frac{7}{8}$	122	314	120	881	126	131	177	170	110	7.40

[a] Proportions given in order of Hydrated Lime : Cement : Sand : Diatomaceous Silica.

TABLE IV.—STRENGTH, PLASTICITY AND COST FIGURES; BASIC 1:3 LIME-SAND RATIO GROUP.

Each result is average of three determinations.

	Mix by Volume[a]	Plasticity Figure	Compressive Strength				Tensile Strength				Cost per Cubic Yard of Wet Mortar, dollars
			28 Days		6 Months		28 Days		6 Months		
			lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	
No. A7	$1:\frac{1}{6}:3:0$	82	72	100	124	100	23	100	57	100	5.82
No. B7	$\frac{3}{4}:\frac{1}{6}:3:\frac{1}{2}$	86	119	165	285	230	25	109	81	142	5.84
No. C7	$\frac{1}{2}:\frac{1}{6}:3:1$	81	221	307	393	317	41	178	71	125	5.87
No. D7	$\frac{1}{4}:\frac{1}{6}:3:1\frac{1}{2}$	81	231	321	445	359	44	191	56	98	5.84
No. A8	$1:\frac{1}{3}:3:0$	96	96	100	297	100	48	100	100	100	6.43
No. B8	$\frac{3}{4}:\frac{1}{3}:3:\frac{1}{2}$	109	160	167	611	206	62	129	117	117	6.45
No. C8	$\frac{1}{2}:\frac{1}{3}:3:1$	104	189	197	622	209	64	133	119	119	6.52
No. D8	$\frac{1}{4}:\frac{1}{3}:3:1\frac{1}{2}$	105	197	205	612	206	68	142	100	100	6.47
No. A9	$1:\frac{1}{2}:3:0$	104	243	100	746	100	88	100	176	100	7.05
No. B9	$\frac{3}{4}:\frac{1}{2}:3:\frac{1}{2}$	123	294	121	926	124	107	122	199	113	7.18
No. C9	$\frac{1}{2}:\frac{1}{2}:3:1$	108	338	139	957	128	111	126	167	95	7.14
No. D9	$\frac{1}{4}:\frac{1}{2}:3:1\frac{1}{2}$	117	336	138	1000	134	129	147	202	115	7.12

[a] Proportions given in order of Hydrated Lime : Cement : Sand : Diatomaceous Silica.

TABLE V.—STRENGTH, PLASTICITY AND COST FIGURES; BASIC 1:4 LIME-SAND RATIO GROUP.

Each result is average of three determinations.

	Mix by Volume[a]	Plasticity Figure	Compressive Strength				Tensile Strength				Cost per Cubic Yard of Wet Mortar, dollars
			28 Days		6 Months		28 Days		6 Months		
			lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	
No. A10	$\frac{3}{4}:\frac{1}{6}:3:0$	61	80	100	122	100	18	100	58	100	5.23
No. B10	$\frac{9}{16}:\frac{1}{6}:3:\frac{3}{8}$	68	120	150	255	209	27	150	66	114	5.30
No. C10	$\frac{3}{8}:\frac{1}{6}:3:\frac{3}{4}$	60	171	214	332	272	32	178	62	107	5 29
No. D10	$\frac{3}{16}:\frac{1}{6}:3:1\frac{1}{8}$	60	190	238	349	286	36	200	43	74	5.36
No. A11	$\frac{3}{4}:\frac{1}{3}:3:0$	73	140	100	482	100	43	100	113	100	5.98
No. B11	$\frac{9}{16}:\frac{1}{3}:3:\frac{3}{8}$	72	177	126	629	130	41	95	110	97	6.05
No. C11	$\frac{3}{8}:\frac{1}{3}:3:\frac{3}{4}$	73	192	137	638	132	42	98	92	81	6.05
No. D11	$\frac{3}{16}:\frac{1}{3}:3:1\frac{1}{8}$	71	211	151	589	122	69	160	116	103	6.08
No. A12	$\frac{3}{4}:\frac{1}{2}:3:0$	74	318	100	801	100	85	100	176	100	6.69
No. B12	$\frac{9}{16}:\frac{1}{2}:3:\frac{3}{8}$	80	328	103	907	113	96	113	231	131	6.74
No. C12	$\frac{3}{8}:\frac{1}{2}:3:\frac{3}{4}$	80	344	108	1020	127	114	134	222	126	6.77
No. D12	$\frac{3}{16}:\frac{1}{2}:3:1\frac{1}{8}$	85	342	108	935	117	114	134	188	107	6.72

[a] Proportions given in order of Hydrated Lime : Cement : Sand : Diatomaceous Silica.

where the percentage increases in strength are small. The mixes employed do not allow many exact comparisons of the effects of straight additions of diatomaceous silica to mixes of constant lime and cement-sand ratios. The results in general are similar to those shown in Table VII, namely a large increase in plasticity and strength in the mixes containing lower proportions of cement and a large increase in plasticity with comparatively little increase in strength in the richer mixes.

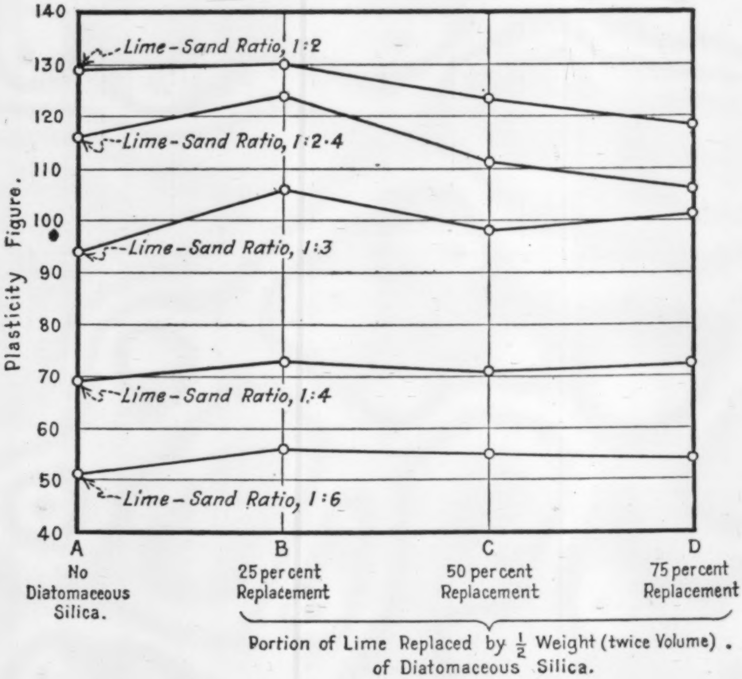

FIG. 4.—Average Plasticity of Groups and Effect of Lime Replacement.

It is believed that the increased strengths obtained with diatomaceous silica are caused by a chemical combination between the hydrated lime, including that formed by the hydrolysis of the cement, and the diatomaceous or amorphous silica. The results show that reduction in the proportion of hydrated lime cannot account for the increased strengths obtained, nor can they be ascribed to a reduction in the water content, as Table VIII shows that the water content is somewhat increased as the hydrated lime is replaced by diatomaceous silica. The reaction between hydrated lime and silica at higher temperatures is well known and is made use of industrially in the manufacture of sand-lime brick. This reaction, which forms monocalcium silicate, has been studied by a number of investigators.[1] The reaction at room temperature takes place much more slowly, but definite evidence of chemical combination has been observed. LeChatelier has prepared and studied the hydrated calcium silicate obtained by

TABLE VI.—STRENGTH, PLASTICITY AND COST FIGURES; BASIC 1:6 LIME-SAND-RATIO GROUP.

Each result is average of three determinations.

	Mix by Volume[a]	Plasticity Figure	Compressive Strength				Tensile Strength				Cost per Cubic Yard of Wet Mortar, dollars
			28 Days		6 Months		28 Days		6 Months		
			lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	
No. A13	$\frac{1}{2}:\frac{1}{6}:3:0$	46	65	100	112	100	16	100	43	100	4.56
No. B13	$\frac{3}{8}:\frac{1}{6}:3:\frac{1}{4}$	50	125	192	192	171	25	156	43	100	4.68
No. C13	$\frac{1}{4}:\frac{1}{6}:3:\frac{1}{2}$	50	133	205	226	202	23	144	33	77	4.64
No. D13	$\frac{1}{8}:\frac{1}{6}:3:\frac{3}{4}$	47	72	111	226	202	38	238	58	135	4.67
No. A14	$\frac{1}{2}:\frac{1}{3}:3:0$	51	152	100	363	100	37	100	118	100	5.41
No. B14	$\frac{3}{8}:\frac{1}{3}:3:\frac{1}{4}$	56	157	103	548	151	32	86	98	83	5.40
No. C14	$\frac{1}{4}:\frac{1}{3}:3:\frac{1}{2}$	56	182	120	609	168	47	127	119	101	5.44
No. D14	$\frac{1}{8}:\frac{1}{3}:3:\frac{3}{4}$	51	193	127	546	150	69	186	117	99	5.54
No. A15	$\frac{1}{2}:\frac{1}{2}:3:0$	57	382	100	1037	100	120	100	202	100	6.17
No. B15	$\frac{3}{8}:\frac{1}{2}:3:\frac{1}{4}$	61	429	112	1104	106	129	108	247	122	6.23
No. C15	$\frac{1}{4}:\frac{1}{2}:3:\frac{1}{2}$	59	409	107	1194	115	130	108	231	114	6.20
No. D15	$\frac{1}{8}:\frac{1}{2}:3:\frac{3}{4}$	64	402	105	1242	120	146	122	252	125	6.18

[a] Proportions given in order of Hydrated Lime : Cement : Sand : Diatomaceous Silica.

TABLE VII.—EFFECT OF ADDING DIATOMACEOUS SILICA TO MORTARS.

	Mix by Volume[a]	Plasticity Figure	Compressive Strength				Tensile Strength			
			28 Days		6 Months		28 Days		6 Months	
			lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent	lb. per sq. in.	Relative to A, per cent
No. A10	$\frac{3}{4}:\frac{1}{6}:3:0$	61	80	100	122	100	18	100	58	100
No. B7	$\frac{3}{4}:\frac{1}{6}:3:\frac{1}{2}$	86	119	149	285	233	25	139	81	140
No. C1	$\frac{3}{4}:\frac{1}{6}:3:1\frac{1}{2}$	111	157	196	461	378	72	400	103	178
No. A11	$\frac{3}{4}:\frac{1}{3}:3:0$	73	140	100	482	100	43	100	113	100
No. B8	$\frac{3}{4}:\frac{1}{3}:3:\frac{1}{2}$	109	160	114	611	127	62	144	117	104
No. C2	$\frac{3}{4}:\frac{1}{3}:3:1\frac{1}{2}$	130	184	131	635	132	84	195	133	118
No. A12	$\frac{3}{4}:\frac{1}{2}:3:0$	74	318	100	801	100	85	100	176	100
No. B9	$\frac{3}{4}:\frac{1}{2}:3:\frac{1}{2}$	123	294	93	926	116	107	126	199	113
No. C3	$\frac{3}{4}:\frac{1}{2}:3:1\frac{1}{2}$	127	321	101	1024	128	117	138	180	102

[a] Proportions given in order of Hydrated Lime : Cement : Sand : Diatomaceous Silica.

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[1] W. E. Emley, "Manufacture and Properties of Sand-Lime Brick," *Technologic Paper No. 85*, U. S. Bureau of Standards.

TABLE VIII.—PERCENTAGE OF WATER ON WEIGHT OF WET MORTARS.

Lime-Sand Ratio	Mix	Mix by Volume[a]	A. Lime-Cement-Sand Mortar; no Diatomaceous Silica	B. 25 Per Cent Lime Replacement[b]	C. 50 Per Cent Lime Replacement[c]	D. 75 Per Cent Lime Replacement[d]
1:2......	No. 1	1½:⅙:3	18.5	20.1	21.3	23.1
	No. 2	1½:⅓:3	18.8	20.1	21.8	23.7
	No. 3	1½:½:3	18.7	20.1	21.3	23.3
1:2.4.....	No. 4	1¼:⅙:3	18.1	19.2	20.2	22.2
	No. 5	1¼:⅓:3	18.4	19.2	20.4	22.4
	No. 6	1¼:½:3	17.8	19.4	20.6	22.2
1:3......	No. 7	1:⅙:3	17.6	19.0	20.0	21.4
	No. 8	1:⅓:3	17.9	18.6	19.4	21.0
	No. 9	1:½:3	17.8	18.4	19.3	20.6
1:4......	No. 10	¾:⅙:3	18.0	18.3	19.6	20.3
	No. 11	¾:⅓:3	17.3	17.8	18.9	19.3
	No. 12	¾:½:3	16.7	17.1	18.0	19.0
1:6......	No. 13	½:⅙:3	18.0	18.3	19.4	19.3
	No. 14	½:⅓:3	16.7	18.0	18.4	18.9
	No. 15	½:½:3	15.9	16.7	17.2	17.8

[a] Proportions given in order of Hydrated Lime : Cement : Sand

[b] Lime-cement-sand mortar: 25 per cent of lime replaced by one-half its weight of diatomaceous silica.

[c] Lime-cement-sand mortar: 50 per cent of lime replaced by one-half its weight of diatomaceous silica.

[d] Lime-cement-sand mortar: 75 per cent of lime replaced by one-half its weight of diatomaceous silica.

TABLE IX.—WEIGHT PER CUBIC FOOT OF WET MORTAR.

Lime-Sand Ratio	Mix	Mix by Volume[a]	A. Lime-Cement-Sand Mortar; no Diatomaceous Silica	B. 25 Per Cent Lime Replacement[b]	C. 50 Per Cent Lime Replacement[c]	D. 75 Per Cent Lime Replacement[d]
1:2......	No. 1	1½:⅙:3	125.9	124.4	123.3	120.3
	No. 2	1½:⅓:3	125.5	124.2	123.1	120.4
	No. 3	1½:½:3	127.2	126.2	123.9	121.3
1:2.4.....	No. 4	1¼:⅙:3	126.0	125.0	123.3	122.0
	No. 5	1¼:⅓:3	129.9	125.0	123.3	121.0
	No. 6	1¼:½:3	128.5	127.4	125.4	123.2
1:3......	No. 7	1:⅙:3	125.5	123.8	123.5	122.0
	No. 8	1:⅓:3	126.7	125.6	125.0	123.4
	No. 9	1:½:3	128.8	128.3	126.5	125.2
1:4......	No. 10	¾:⅙:3	125.1	124.7	123.6	122.6
	No. 11	¾:⅓:3	127.8	127.0	126.6	124.7
	No. 12	¾:½:3	129.4	128.6	127.6	126.5
1:6......	No. 13	½:⅙:3	123.5	124.4	123.7	122.3
	No. 14	½:⅓:3	125.9	126.0	125.8	126.2
	No. 15	½:½:3	128.6	128.4	127.9	126.5

[a] Proportions given in order of Hydrated Lime : Cement : Sand.

[b] Lime-cement-sand mortar: 25 per cent of lime replaced by one-half its weight of diatomaceous silica.

[c] Lime-cement-sand mortar: 50 per cent of lime replaced by one-half its weight of diatomaceous silica.

[d] Lime-cement-sand mortar: 75 per cent of lime replaced by one-half its weight of diatomaceous silica.

adding an excess of lime-water to a colloidal solution of silicic acid. He considers the compound formed to have the composition $2CaSiO_3 \cdot 5H_2O$.[1, 2]

TABLE X.—BRICK-MORTAR BOND ADHESIVE STRENGTHS AT 28 DAYS.

(Total Force, in pounds, Required to Pull Apart 16 sq. in. Bond.)

Lime-Sand Ratio	Mix	Mix by Volume[a]	A Lime-Cement-Sand Mortar; no Diatomaceous Silica	B 25 Per Cent Lime Replacement[b]	C 50 Per Cent Lime Replacement[c]	D 75 Per Cent Lime Replacement[d]	Average of A, B, C and D
1:2......	No. 1	$1\frac{1}{2}:\frac{1}{6}:3$	131	159	171	238	175
	No. 2	$1\frac{1}{2}:\frac{1}{3}:3$	181	54	225	286	187
	No. 3	$1\frac{1}{2}:\frac{1}{2}:3$	93	135	90	70	97
	Average..	....	135	116	162	198	153
1:2.4....	No. 4	$1\frac{1}{4}:\frac{1}{6}:3$	72	107	148	247	144
	No. 5	$1\frac{1}{4}:\frac{1}{3}:3$	140	214	166	124	161
	No. 6	$1\frac{1}{4}:\frac{1}{2}:3$	177	118	195	165	164
	Average..	....	130	146	170	179	156
1:3......	No. 7	$1:\frac{1}{6}:3$	34	118	236	18	102
	No. 8	$1:\frac{1}{3}:3$	59	284	162	143	162
	No. 9	$1:\frac{1}{2}:3$	218	98	286	216	205
	Average..	....	104	167	228	126	156
1:4......	No. 10	$\frac{3}{4}:\frac{1}{6}:3$	163	267	193	261	221
	No. 11	$\frac{3}{4}:\frac{1}{3}:3$	317	174	187	128	202
	No. 12	$\frac{3}{4}:\frac{1}{2}:3$	361	194	202	231	247
	Average..	....	280	212	194	207	223
1:6......	No. 13	$\frac{1}{2}:\frac{1}{6}:3$	266	288	359	172	271
	No. 14	$\frac{1}{2}:\frac{1}{3}:3$	306	318	378	303	326
	No. 15	$\frac{1}{2}:\frac{1}{2}:3$	591	555	488	567	550
	Average..	....	388	387	408	347	382
	Average of all		208	206	232	211	

[a] Proportions given in order of Hydrated Lime : Cement : Sand.
[b] Lime-cement-sand mortar: 25 per cent of lime replaced by one-half its weight of diatomaceous silica.
[c] Lime-cement-sand mortar: 50 per cent of lime replaced by one-half its weight of diatomaceous silica.
[d] Lime-cement-sand mortar: 75 per cent of lime replaced by one-half its weight of diatomaceous silica.

The results of the adhesion tests show that replacement of the lime with diatomaceous silica does not affect the average adhesive strength. Table X presents these results in detail. It is noteworthy that the average of all the adhesive strengths remains practically constant in the upper three groups of more plastic mixes and then

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[1] J. W. Mellor, "Treatise on Inorganic and Theoretical Chemistry," Vol. VI, p. 359.

[2] H. LeChatelier, "Kieselsäure und Silicate," p. 337 (1920).

TABLE XI.—COST, DOLLARS PER CUBIC YARD OF WET MORTAR.[a]

Lime-Sand Ratio	Mix	Mix by Volume[b]	A. Lime-Cement-Sand Mortar; no Diatomaceous Silica	B. 25 Per Cent Lime Replacement[c]	C. 50 Per Cent Lime Replacement[d]	D. 75 Per Cent Lime Replacement[e]
1:2......	No. 1	1½:⅙:3	6.87	6.81	6.87	6.84
	No. 2	1½:⅓:3	7.24	7.31	7.32	7.20
	No. 3	1½:½:3	7.78	7.85	7.85	7.73
1:2.4.....	No. 4	1¼:⅙:3	6.33	6.40	6.42	6.44
	No. 5	1¼:⅓:3	7.04	6.95	6.94	6.85
	No. 6	1¼:½:3	7.53	7.55	7.51	7.40
1:3......	No. 7	1:⅙:3	5.82	5.84	5.87	5.84
	No. 8	1:⅓:3	6.43	6.45	6.52	6.47
	No. 9	1:½:3	7.06	7.18	7.14	7.12
1:4......	No. 10	¾:⅙:3	5.23	5.30	5.29	5.36
	No. 11	¾:⅓:3	5.98	6.05	6.05	6.08
	No. 12	¾:½:3	6.69	6.74	6.77	6.72
1:6......	No. 13	½:⅙:3	4.56	4.68	4.64	4.67
	No. 14	½:⅓:3	5.41	5.40	5.44	5.54
	No. 15	½:½:3	6.17	6.23	6.20	6.18

[a] Costs are based on the following Los Angeles prices: Sand, $1.75 per ton, including 5-per-cent moisture; Hydrated Lime, $18.00 per ton; Cement, $2.60 per bbl.; Diatomaceous Silica (Celite), $40.00 per ton.

[b] Proportions given in the order of Hydrated Lime : Cement : Sand.

[c] Lime-cement-sand mortar: 25 per cent of lime replaced by one-half its weight of diatomaceous silica.

[d] Lime-cement-sand mortar: 50 per cent of lime replaced by one-half its weight of diatomaceous silica.

[e] Lime-cement-sand mortar: 75 per cent of lime replaced by one-half its weight of diatomaceous silica.

TABLE XII.—TIME OF SET.

		Initial Set, hours				Final Set, hours			
	Mix by Volume[a]	A. Lime-Cement-Sand Mortar; no Diatomaceous Silica	B. 25 Per Cent Lime Replacement[b]	C. 50 Per Cent Lime Replacement[c]	D. 75 Per Cent Lime Replacement[d]	A. Lime-Cement-Sand Mortar; no Diatomaceous Silica	B. 25 Per Cent Lime Replacement[b]	C. 50 Per Cent Lime Replacement[c]	D. 75 Per Cent Lime Replacement[d]
No. 1	1½:⅙:3	52	48	47	38	81	84	84	86
No. 2	1½:⅓:3	18	21	21	26	26	32	33	33
No. 3	1½:½:3	10	10	12	12	23	24	25	26
No. 4	1¼:⅙:3	51	46	38	30	99	94	86	75
No. 5	1¼:⅓:3	18	22	19	24	30	35	32	33
No. 6	1¼:½:3	10	11	10	9	22	24	21	24
No. 7	1:⅙:3	35	42	28	30	65	72	42	50
No. 8	1:⅓:3	23	22	18	18	35	34	34	32
No. 9	1:½:3	12	12	11	8	26	25	25	21
No. 10	¾:⅙:3	33	31	37	27	60	58	54	42
No 11	¾:⅓:3	15	17	16	13	30	31	30	23
No. 12	¾:½:3	10	8	7	7	21	22	22	21
No. 13	½:⅙:3	40	31	30	34	64	72	54	68
No. 14	½:⅓:3	16	14	14	11	26	28	25	27
No. 15	½:½:3	8	8	10	10	21	20	20	22

[a] Proportions given in order of Hydrated Lime : Cement : Sand.

[b] Lime-cement-sand mortar: 25 per cent of lime replaced by one-half its weight of diatomaceous silica.

[c] Lime-cement-sand mortar: 50 per cent of lime replaced by one-half its weight of diatomaceous silica.

[d] Lime-cement-sand mortar: 75 per cent of lime replaced by one-half its weight of diatomaceous silica.

very markedly increases in the two less plastic mixes, being highest of all in the leanest and least plastic mixes. Also, in the plastic mixes and in the less plastic 1:4 lime-sand group, there seems to be very little increase in adhesive strength as the proportion of cement is increased. In the 1:3 and 1:6 lime-sand groups, however, there seems to be a regular increase in adhesive strength as the proportion of cement is increased.

It is felt that the modified method of determining plasticity has produced results correctly expressing this property. Considerable time was spent in modifying the procedure to make it suitable for expressing the plasticity of mortars. The results obtained, although not very precise, agree well with the apparent plasticity or "feel" of a mortar when worked with a trowel. The plasticity figure does not, however, give an expression of the time during which a mortar containing cement remains workable. As the proportion of cement is increased, the plasticity, as is evident from working with a trowel and from the plasticity figure, increases, but the mix starts to set more quickly and does not retain its workable properties for as long a period as a mix with less cement.

The *addition* of diatomaceous silica to a mortar increases its plasticity as well as its strength. The proportional *replacement* of lime with diatomaceous silica, according to the plan here followed, gives mixes of practically equal plasticity and cost, but with greatly increased strengths. The average plasticity of the groups is graphically shown in Fig. 4.

These results show that lime-cement mortars are improved by the use of diatomaceous silica. The use of a greater ratio of diatomaceous silica to hydrated lime than corresponds to 50 per cent replacement ("C" mixes) is not recommended with the type of mortar mixes here considered. A greater proportion of diatomaceous silica may properly be used if sufficient lime is also present to correspond to the proportions in C mixes, whenever it is desired to increase the plasticity as well as the strength, although this will increase the cost.

The advantages of using diatomaceous silica as an admixture in concrete have been dealt with in a number of technical papers during the last several years. It would seem that the possibility of employing diatomaceous silica as an admixture for mortars holds similar promise and it is hoped that the data herein presented will suggest further work along similar lines by others interested in both the laboratory and field aspects of building construction.

DISCUSSION

Mr. Smith.

MR. G. A. SMITH[1] (*presented in written form*).—A supplementary series of tests was made on basic 1:3 cement-sand ratio mortars using the same materials and methods described in Mr. Weymouth's paper. Such mortars are in common use and cover a range of proportions not included in the paper. These tests were made to study the effect of adding hydrated lime and diatomaceous silica to mortars richer in cement than those reported in Mr. Weymouth's paper.

Fourteen different mixes were used, which contained 5 and 10 per cent of hydrated lime based on the weight of cement, and admixtures of diatomaceous silica varying from 0 to 6 per cent of the weight of cement. The plasticities and 28-day strengths are shown in Table I.

TABLE I.—RESULTS OF TESTS ON BASIC 1:3 CEMENT-SAND MORTARS.

Admixture, per cent[a]		Plasticity Figure	28-Day Compressive Strength		28-Day Tensile Strength		Weight per Cubic Foot, lb.	Water		Cost per Cubic Yard, dollars
Hydrated Lime	Diatomaceous Silica		lb. per sq. in.	Per Cent of Mixture with no Diatomaceous Silica	lb. per sq. in.	Per Cent of Mixture with no Diatomaceous Silica		Per Cent of Dry Materials	Water-Cement Ratio	
5....	0	82	2262	100	308	100	126.0	18.0	1.161	6.98
	1	92	2266	100	346	112	126.4	18.2	1.172	7.11
	2	113	2044	90	344	112	126.9	18.5	1.197	7.22
	3	123	2515	111	368	119	126.9	18.5	1.197	7.36
	4	118	2423	107	384	125	127.1	18.8	1.219	7.46
	5	119	2453	108	370	120	126.4	19.3	1.257	7.51
	6	131	2079	92	332	108	126.4	20.2	1.316	7.57
10...	0	94	2240	100	347	100	125.9	18.2	1.186	7.18
	1	110	2450	109	380	110	128.6	17.1	1.113	7.52
	2	116	2455	109	394	114	128.2	17.7	1.169	7.58
	3	116	2300	103	369	106	127.3	18.5	1.211	7.60
	4	114	2423	108	371	107	127.6	18.7	1.224	7.71
	5	118	2273	101	357	103	127.4	19.0	1.253	7.79
	6	118	2328	104	372	107	126.8	19.4	1.281	7.84

[a] Admixtures, per cent by weight of cement.

The diatomaceous silica produces a marked increase in plasticity up to 3 per cent addition. Further additions up to 6 per cent cause no further improvement in plasticity. The addition of diatomaceous silica has very little effect upon the compressive strength with 5 per cent of lime but the tensile strength is noticeably increased. The addition of diatomaceous silica produces some increase in both compressive and tensile strength in the mixes containing 10 per cent of lime.

Practical tests in laying brick confirm the plasticity increases shown in these mixes. It is believed that the saving in labor due to this increased plasticity (with diatomaceous silica admixtures up to 3 per cent) more than offsets the increased cost.

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[1] Concrete Engineer, Celite Products Co., Lompoc, Calif.

THE RECOVERY AND EXAMINATION OF THE ASPHALT IN ASPHALTIC PAVING MIXTURES

By John H. Bateman[1] and Charles Delp[2]

Synopsis

This paper describes a method for extracting and recovering the asphalt in asphaltic paving mixtures by which the physical characteristics of the asphalt are not materially changed. The method makes use of common processes of extraction of the asphalt but introduces a modification of the usual procedure for distilling off the solvent in the recovery of the asphalt. By the use of a partial vacuum, distillation may be carried out without materially affecting the physical characteristics of the asphalt in the condition in which it is present in the paving mixture.

Studies have shown that the process of mixing asphalt with mineral aggregates causes oxidation of the asphalt. The extent of this oxidation is shown by comparative tests of asphalt prior to and subsequent to mixing.

The method as described is applicable to the examination of the bitumen in rock asphalts.

Introduction

Proper laboratory control of the composition and preparation of asphaltic paving mixtures requires the analysis of the mixtures to determine the bitumen content and the proportions of various sizes of the mineral aggregate present in the mixtures. It is the usual practice in laboratory procedure to extract the bitumen from these mixtures by dissolving it in carbon bisulfide (with the aid of a centrifuge), the mineral aggregate being left after all the bitumen has been removed. This procedure enables a quantitative determination of the bitumen to be made. Laboratory operators have long desired a method by which the dissolved bitumen might be recovered from the solution in carbon bisulfide so that physical tests of the bitumen itself might be made. A method of recovery which would not affect the physical properties of the bitumen would be useful in many respects; it would enable determinations to be made of the effect of the plant preparation and of the mixing operation on the physical characteristics of the bitumen, thus providing a practicable means of determining whether, in any instance, the bitumen has been damaged; it would provide a means of determining, within a reasonable degree of accuracy, the character of the bitumen in its original condition before being incorporated in the paving mixture; and it would be useful in connection with studies of the bitumen contained in rock asphalts. It would also enable the development of many research investigations in connection with the construction of asphaltic pavements.

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[1] Research Professor of Highway Engineering, Louisiana State University, Baton Rouge, La.

[2] Chemist, Louisiana Highway Commission, Baton Rouge, La.

The recovery of the asphalt from paving mixtures involves two separate operations: the extraction of the asphalt from the mixture by means of a solvent, and the removal of the solvent. The extraction operation may be most conveniently and efficiently performed by means of a centrifugal extractor. The usual methods of recovering the asphalt consist of evaporation[1] or distillation of the solvent. The latter process is desirable in order to recover the solvent and thereby effect an economy. These two processes, as they have ordinarily been conducted, possess two serious objections: first, the complete removal of the solvent is very difficult; and second, the effect of solvents on the physical properties of asphalt has not been fully known. In addition to these two features of the problem, another unknown factor is present, namely, the effect of the mixing operation on the asphalt. It has been generally recognized that asphalt extracted from a paving mixture has different physical characteristics than those of the asphalt prior to the mixing operation, but the extent of each of the previously described influences has been unknown. The authors have for some time been conducting an investigation which had for its purpose the determination of these influences and have developed a method of recovering the asphalt which gives promise of accomplishing the desired results.

EXTRACTION OF THE ASPHALT

The extraction of the asphalt from asphaltic mixtures was performed with a Rotarex centrifugal extractor of 1000 g. capacity. Sheet asphalt was the only artificial mixture studied and a sufficient quantity of each mixture was used to obtain approximately one pound of recovered asphalt. Carbon bisulfide was the solvent used in the tests which will be described. Some work was done with two grades of benzol but this solvent was not completely studied. It is extremely difficult, if not impossible, to prevent some very finely-divided mineral matter from coming through the filter pads of a centrifugal extractor and in our work we found it necessary to remove this mineral matter by sedimentation and further filtration. The solution was placed in large stoppered glass bottles which were allowed to stand, without disturbance, for several days. The solution was then carefully decanted, by siphoning, to within one or two inches of the bottom of each bottle. The decanted solution was then filtered through Gooch crucibles (of the type and size used for solubility tests of asphalt), using an asbestos mat. The Gooch crucibles were operated in pairs, the suction being obtained by means of a filter pump attached to a water outlet. The crucibles became clogged in spite of the mineral matter removed by sedimentation and several extra crucibles were provided to enable cleaning of the clogged crucibles without any cessation of the filtering process. A tube centrifuge might be used to remove the mineral matter in place of sedimentation but a very large quantity of solution must be handled which is an objection to its use.

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[1] A. W. Dow, "The Testing of Bitumens for Paving Purposes," *Proceedings*, Am. Soc. Testing Mats., Vol. III, p. 349 (1903).

REMOVAL OF THE SOLVENT

The removal of the solvent was carried out by vacuum distillation. The filtered solution was placed in a large distillation flask of 2000-cc. capacity and direct heat applied by means of a Bunsen burner, the carbon bisulfide being collected in a receptacle provided for that purpose.

The temperature of the asphalt solution cannot be too rapidly increased during the early stages of the distillation process on account of the foaming of the solution. The temperature was, however, slowly increased until 300° F. was reached, at which point only a small amount of the solvent remained in the flask and foaming had ceased. The distillation flask was then connected with a filter pump (attached to a water outlet) and distillation was continued with the aid of a partial vacuum (180 mm. of mercury) raising the temperature to 500° F.[1] at the rate of 5° F. per minute. The heat was then removed immediately and the asphalt poured into penetration cans, ductility molds, softening point rings, or other testing devices.

OUTLINE OF TESTS

Extensive tests were carried out to establish the accuracy of the method and to determine the effect of the solvent and of the mixing process on the physical properties of petroleum asphalt. All of the

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[1] A maximum temperature of 425° F. was used in the recovery of the asphalt having an original penetration of 214. This temperature is recommended for asphalts having an original penetration (at normal temperature) greater than 150.

TABLE I.—TESTS OF PETROLEUM ASPHALT, 36 PENETRATION.

Test	Characteristics of the Original Asphalt	Characteristics of the Asphalt after Recovery from Solution in Carbon Bisulfide		Characteristics of the Asphalt after Recovery from a Mixture with Mineral Aggregate
		By Straight Distillation of the Solvent	By Vacuum Distillation of the Solvent	
Specific gravity 77°/77° F	1.051	1.051	1.051	1.054
Flash point, open cup, deg. Fahr	560	500	560	565
Fire point, open cup, deg. Fahr	685	680	685	685
Softening point, Ring and Ball Method, deg. Fahr.	140	138	140	153
Penetration at 39° F	19	26	20	15
Penetration at 77° F	36	41	36	25
Penetration at 115° F	126	166	126	77
Ductility at 77° F	100+	100+	100+	20
Loss at 325° F., 50 g., 5 hrs	0.09	0.29	0.09	0.09
Penetration of residue at 77° F	32	28	31	21
Bitumen (solubility in CS_2), per cent	99.73	99.87	99.79	99.86
Per cent of bitumen soluble in CCl_4	99.94	99.96	99.95	99.99
Per cent of bitumen soluble in 86° Be, naphtha	68.56	68.02	68.26	63.23
Sulfur, per cent	6.50	6.48	6.47	6.17

TABLE II.—TESTS OF PETROLEUM ASPHALT, 119 PENETRATION.

Test	Characteristics of the Original Asphalt	Characteristics of the Asphalt after Recovery from Solution in Carbon Bisulfide		Characteristics of the Asphalt after Recovery from a Mixture with Mineral Aggregate
		By Straight Distillation of the Solvent	By Vacuum Distillation of the Solvent	
Specific gravity 77°/77° F	1.039	1.038	1.039	1.044
Flash point, open cup, deg. Fahr	485	455	480	490
Fire point, open cup, deg. Fahr	600	590	600	605
Softening point, Ring and Ball Method, deg. Fahr.	113	112	113	134
Penetration at 39° F	54	60	51	33
Penetration at 77° F	119	127	118	57
Penetration at 115° F	too soft	too soft	too soft	212
Ductility at 77° F	100+	100+	100+	40
Loss at 325° F., 50 g., 5 hrs	0.28	0.39	0.26	0.24
Penetration of residue at 77° F	88	90	87	50
Bitumen (solubility in CS_2), per cent	99.72	99.85	99.82	99.79
Per cent of bitumen soluble in CCl_4	99.83	99.89	99.85	99.85
Per cent of bitumen soluble in 86° Be, naphtha	69.44	69.39	69.43	68.10
Sulfur, per cent	6.28	6.28	6.26	5.91

TABLE III.—TESTS OF PETROLEUM ASPHALT, 214 PENETRATION.

Test	Characteristics of the Original Asphlat	Characteristics of the Asphalt after Recovery from Solution in Carbon Bisulfide		Characteristics of the Asphalt after Recovery from a Mixture with Mineral Aggregate
		By Straight Distillation of the Solvent	By Vacuum Distillation of the Solvent	
Specific gravity 77°/77° F	1.032	1.032	1.032	1.036
Flash point, open cup, deg. Fahr	430	410	430	440
Fire point, open cup, deg. Fahr	540	540	540	550
Softening point, Ring and Ball Method, deg. Fahr.	100	98	100	110
Penetration at 39° F	98	102	95	88
Penetration at 77° F	214	230	216	161
Penetration at 115° F	too soft	too soft	too soft	too soft
Ductility at 77° F	too soft	too soft	too soft	too soft
Loss at 325° F., 50 g., 5 hrs	0.50	0.88	0.51	0.52
Penetration of residue at 77° F	152	141	155	116
Bitumen (solubility in CS_2), per cent	99.87	99.94	99.94	99.89
Per cent of bitumen soluble in CCl_4	99.94	99.98	99.97	99.96
Per cent of bitumen in 86° Be, naphtha	74.19	74.01	74.06	72.92
Sulfur, per cent	6.23	6.15	6.20	5.98

petroleum asphalt used in the investigation was refined from Mexican petroleum, and consisted of three commercial grades having at normal temperature original penetrations of 36, 119, 214. The following procedure was followed in making tests of the petroleum asphalt:

1. The characteristics of the several asphalts were determined.

2. Each asphalt was dissolved in carbon bisulfide and the asphalt recovered by

(*a*) Straight distillation of the solvent; and

(*b*) Vacuum distillation of the solvent according to the method herein described.

3. Each asphalt was mixed with sand and limestone dust to produce a typical sheet asphalt mixture. The asphalt was then extracted from each mixture and recovered by vacuum distillation of the solvent according to the method herein described.

TABLE IV.—TESTS OF PETROLEUM ASPHALT EXTRACTED FROM A PLANT SAMPLE OF ASPHALTIC CONCRETE.

Test	Characteristics of the Original Asphalt	Characteristics of the Asphalt after Extraction and Recovery by Vacuum Distillation of the Solvent
Specific gravity 77°/77° F.	1.046	1.053
Flash point, open cup, deg. Fahr.	520	530
Fire point, open cup, deg. Fahr.	640	650
Softening point, Ring and Ball Method, deg. Fahr.	141	156
Penetration at 39° F.	16	13
Penetration at 77° F.	36	26
Penetration at 115° F.	139	85
Ductility at 77° F.	100+	15
Loss at 325° F., 50 g., 5 hrs.	0.17	0.17
Penetration of residue at 77° F.	31	21

Tests of asphalt recovered from mixtures prepared under field conditions were also made.

DISCUSSION OF TEST RESULTS

Tables I, II and III give the results of tests on the three asphalts according to the outline described. Table IV gives the results of tests on an asphalt mixed with mineral aggregate under field conditions.

Effect of Ordinary Distillation.—Reference to the second columns of Tables I, II and III shows that ordinary distillation to safe temperatures and within reasonable time limits will not entirely remove the solvent. The presence of some solvent is best shown by the results of the tests for flash point, penetration at 115° F., and loss on heating. It will be observed that the change in the characteristics of the asphalts indicates merely a softening effect due to the presence of some solvent.

Effect of the Solvent.—Examination of the third columns of Tables I, II and III indicate that no appreciable change in the physical characteristics of the asphalt has been caused by the carbon bisulfide. A slight reduction in the sulfur content was obtained in all cases where the asphalt was in solution with the solvent.

Effect of the Mixing Process.—A careful examination of the test results in the fourth columns of Tables I, II and III, and the second column of Table IV, reveals the fact that asphalt which has been mixed with mineral aggregate has undergone a marked physical change in the following respects:

1. The specific gravity was slightly increased.
2. The flash point and fire point were slightly increased.
3. The softening point was materially increased.
4. The penetration at all temperatures was materially lowered and the penetrations at low and high temperatures were drawn more closely together.
5. The ductility of the two harder asphalts was greatly reduced.

All of these changes in physical properties indicate that oxidation had taken place in the mixing process. An excellent opportunity for oxidation of the asphalt is present during mixing on account of the great surface area of hot asphalt which is exposed to the air on the surface of the particles of the mineral aggregate.

CONCLUSIONS

The following conclusions are drawn:

1. The recovery of asphalt from solution in carbon bisulfide may be accomplished by vacuum distillation of the carbon bisulfide, without an appreciable effect of the solvent on the physical characteristics of the asphalt.
2. Asphalt refined from Mexican petroleum is oxidized during the process of mixing with a mineral aggregate and its characteristics are therefore changed. The extent of this change may be determined by examination of the material extracted with carbon bisulfide and recovered by vacuum distillation of the solvent.

DISCUSSION

Mr. C. N. Forrest.[1]—I read this paper with considerable interest and think it is a timely offering to the subject. The extraction of bitumen from paving and other bituminous mixtures, so that the extracted matter may be subsequently examined and valued, is an important matter. It seems to me that the authors understand what they are after. They have presented supporting data for their conclusions, and I think, as I said before, it is a very worthy contribution to our knowledge of this particular art and the authors are to be congratulated on their work. **Mr. Forrest.**

Mr. W. Furber Smith.[2]—The authors point to the suitability of this method for the examination of rock asphalts. It has been the experience of our laboratory that, in the examination of rock asphalts, several difficulties are encountered which are inconsequential in the extraction of refined asphalts. Rock asphalts quite often contain small amounts of bituminous material volatile at very low temperatures. The removal of such material often produces a hardening of the residual material out of all proportion to the loss by weight. In working with such materials, every method we have tried, including straight and vacuum distillation and prolonged evaporation in an oven at very low temperatures (with a complete series of control samples of varying original consistencies), has resulted in a residue of as low as 20 penetration in certain instances where the original material was very soft (say 200 to 300 penetration). There is a very real question as to the applicability of this method or any other that has been advanced to date to the extraction for examination of bitumens of this type. **Mr. Smith.**

Mr. J. S. Miller.[3]—I endorse everything Mr. Forrest has said regarding this paper. There is one point, however, that I think should be given consideration by the Society in the publication of papers of this kind. When the reports of tests are out of line with the standards established by the Society—I have in mind particularly a penetration at 39° F., instead of at 32° F.—they cannot be correlated with previous results of other workers. It is an unfortunate circumstance. **Mr. Miller.**

The Chairman (*Mr. K. G. Mackenzie*[4]).—I should like to see, if possible, some sort of a regulation whereby our methods would **Mr. Mackenzie.**

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[1] The Barber Asphalt Co., Philadelphia, Pa.

[2] Assistant Testing Engineer, Oklahoma State Highway Commission, Oklahoma City, Okla.

[3] Director, Technical Bureau, The Barber Asphalt Co., Maurer, N. J.

[4] Consulting Chemist, The Texas Co., New York City.

Mr. Mackenzie. have to be used; but the unfortunate part is, I assume, that a definite piece of research work is completed not using our standard methods, and we have the choice of accepting or rejecting it. I really feel that this is part of the general educational campaign in which we are all interested, in trying to secure the universal adoption of our methods. If you have any suggestions, Mr. Miller, as to how this might be straightened out, I should be glad to entertain a motion for its consideration by the proper body within the Society.

Mr. Miller. MR. MILLER.—Probably Committee E-6 on Papers and Publications would be the proper body and I should like to move that this matter be called to the attention of that committee with the request that the committee consider the possibility of calling the attention of all authors of papers presented before the Society to the desirability of reporting test results wherever possible in accordance with the standard methods adopted by the Society.

[The motion was seconded and unanimously adopted.—ED.]

Mr. Pullar. MR. H. B. PULLAR.[1]—I notice that one of the conclusions of the paper is that "Asphalt refined from Mexican petroleum is oxidized during the process of mixing with a mineral aggregate and its characteristics are therefore changed." I do not think that the author has given any satisfactory evidence to support such a conclusion. The change in ductility from over 100 cm. to 15 cm., as stated on page 469, and the low susceptibility to changes of temperature have evidently been the reason for this conclusion, but I do not think that these tests on an extracted bitumen by the method described are positive proof of oxidation taking place during the mixing. My own experience in the so-called oxidation of petroleum oils would not warrant any such conclusions from the results obtained.

Mr. Dow. MR. A. W. DOW.[2]—I beg to differ with Mr. Pullar. It is my experience that asphalts are readily oxidized when exposed in thin films to the air even for a short time and at a temperature as low as 300° F. Mr. Hemstreet of the Hastings Paving Co. patented a device which I have seen in operation. This consists of a heated cylinder partly immersed in an oil tank. As this cylinder revolves, the thin film of oil exposed on its surface is oxidized by the air. By this means one can oxidize the entire tank of oil. This oxidation readily takes place at a temperature of 350° F.

Mr. Miller. MR. MILLER.—I agree more or less with certain portions of Mr. Pullar's comments and his conclusions. I do think that the results indicate that some change has occurred, whether it is oxidation or not is left to the author to tell us his opinion. I do not believe that oxidation will occur with the time and temperature present in the paving mixer, but I do think there is a possibility of a reaction with sulfur at that temperature and in that time cycle. Of course it may be the author of this paper has not considered that phase, but that is possibly the reason for this change in the material. **Mr. Miller.**

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[1] Secretary and Manager, Heavy Oil Sales, James B. Berry's Sons Co., Chicago, Ill.

[2] Dow and Smith, Paving and Chemical Engineers, New York City.

THE CHAIRMAN.—I do not wish to speak from the chair, but Mr. Miller has covered the very point I had in mind, that it does seem that the question of some chemical reaction with the carbon bisulfide evidently has not been considered. **Mr. Mackenzie.**

MR. MILLER.—I did not mean carbon bisulfide, because the same effect can be noted by making the extraction with benzol. It is unfortunate the authors did not use benzol. I think they ran an extraction of bitumen without making a mixture prior to the extraction with bisulfide, to show that the sulfur in the bisulfide was not causing this reaction. **Mr. Miller.**

MR. FORREST.—I wish to add one thought more on this particular subject. The term oxidation, as used in this article, is generic, I think, and may reasonably be enlarged to include polymerization as well as oxidation. While all of the changes in the bitumens which have been disclosed by this investigation may not be due solely to oxidation in the strict sense of the word, these are the same changes that are realized in the art of air treating petroleum products which are designed primarily from an oxidation point of view and cause a modification in melting point and ductility of bitumen such as the authors have shown to be caused by the manipulation of asphalt in the ordinary paving and mixing operation without intentional air blowing. **Mr. Forrest.**

MR. DOW.—There is one thing I wish to call to your attention which rather discounts Mr. Miller's suggestion that the changes might be due to sulfur. If the asphalt is heated in bulk, there is no such change that takes place even when the temperatures are carried much higher than that stated. You must appreciate that there is a tremendous area of asphalt cement exposed on the surface of sand grains when the materials are being mixed, in fact many hundred times the area as is exposed in Mr. Hemstreet's apparatus. **Mr. Dow.**

MR. MILLER.—I think that any one who has worked with a series of asphalts from different sources and has gone after this one particular point will realize that one cannot get an oxidation reaction or an effect indicating some sort of reaction or polymerization on all sorts of asphalt under this same time cycle, and that is one reason why I have felt for some time that this reaction was very closely **Mr. Miller.**

Mr. Miller. related to sulfur, because it does not occur in all classes of asphalt starting from the same penetration, putting it through the same operation of extraction with the same solvents and recovering the extracted bitumen for examination.

Mr. Pullar. MR. PULLAR.—My experience confirms, to a great extent, Mr. Forrest's and Mr. Miller's statements in respect to the action of air on residuum oils, and I am of the opinion that the reaction taking place by the treatment of residuum oil with air is principally a polymerizing action rather than oxidation. I do not believe that it is possible to get an oxidizing reaction at the temperature of a paving mixture, nor do I think it possible to get the changes, as shown by the results of this test, by any such operation, and I doubt very much whether there was any oxidizing effect prior to the laying of the finished paving mixture. In all the patent litigation over blown asphalts, there has never been any conclusive evidence to show that there was a fixation of oxygen or a combination of hydrogen and oxygen in the form of water due to the reaction of blowing the hot oil with air, and I am strongly of the opinion that the results obtained by the author were due to other causes than those stated.

Mr. Smith. MR. SMITH.—Mr. Miller suggests that the reaction described by the authors as oxidation may be due to sulfur. I do not quite understand when this reaction with sulfur is thought to have occurred. I should like to ask if Mr. Miller had in mind a possible reaction with the sulfur normally contained and prior to extraction, or was it thought that sulfur resulting from the decomposition of the solvent produced the observed results during the extraction process?

Mr. Miller. MR. MILLER.—I think the data presented by the author clearly proves that the sulfur reaction does not proceed from the solvent used, and I have never thought that sulfur from any other source was necessary, but it is the inherent sulfur content of the oil in its peculiar chemical combination that may be a factor in this. Whether it is oxidation or polymerization I do not know, but I think it is closely related to sulfur.

Mr. Hubbard. MR. PRÉVOST HUBBARD.[1]—If Mr. Miller's assumptions were correct, then reaction with sulfur would occur, at the temperature of mixing, in the mass as quickly as in the thin film. It seems to me that Mr. Dow's conclusions are much more logical and that there is really an oxidation of the thin films at a somewhat lower temperature than is used for commercial oxidation of asphalt. It is simply a matter of degree; there is more rapid oxidation of masses at higher temperatures, but some oxidation takes place in the thin film at lower

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[1] Chemical Engineer, The Asphalt Association, New York City.

Mr. Hubbard.

temperatures. It seems to me that if sulfur is the cause, the sulfur would have reacted in the masses of hot asphalt the same as in the thin films at a lower temperature.

Mr. Miller.

MR. MILLER.—I think Mr. Hubbard misunderstood my statement. I am wondering if it is not bound up in the presence of the sulfur compound and other things which cause the sulfur to react? I do not pretend for a moment that the sulfur compound or sulfur would react in mass at the temperature and in the time cycle here involved, but these reactive sulfur compounds present in certain oils and the residues produced therefrom, under certain conditions of time, temperature and thickness of film—I think thickness of film is a factor—produce this phenomenon that the author calls oxidation. That is the only reason I raised the question as to whether the term "oxidation" should be used, because I think it is not broad enough.

Mr. Fulweiler.

MR. W. H. FULWEILER.[1]—There are many cases where it is apparent that some form of sulfur seems to be in a very reactive state; whether it acts as a catalyst in causing these changes to take place, I do not know, but I do know that changes do take place in certain cases.

Mr. Church.

MR. S. R. CHURCH.[2]—It has long been a matter of interest to me to know just what is the chemical explanation of the so-called "oxidation" of asphalt oils. It seems to me that the discussion has justified the authors' use of the term. After all, they are stating certain facts, they are not theorizing, and the term is used broadly to describe the changes that take place, which cannot be entirely explained by temperature.

Mr. Smith.

MR. SMITH (*by letter*).—During the discussion of this paper following its presentation at the annual meeting, exception was taken to the authors' use of the term "oxidation" in describing the reaction undergone by the asphaltic cement when mixed with mineral aggregate in the preparation of the paving mixture. The authors' results point very strongly to the similarity of the results of the reaction involved at the mixer to those obtained through the blowing process of refining. This similarity might be anticipated from an analysis of the two processes. In the one case (at the mixer) intimate contact between oxygen of the air and the bitumen is brought about by the exposure of thin films of asphalt with a large excess of air for a short time at a temperature only slightly lower than has been shown[3], [4]

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[1] Chemical Engineer, United Gas Improvement Co., Philadelphia, Pa.

[2] Consulting Chemist, New York City.

[3] Gesner, "A Practical Treatise on Coal Petroleum and Other Distilled Oils," Second Edition (1865), p. 128.

[4] Byerly, U. S. Patent 524,130, August 7, 1894.

Mr. Smith. to be high enough for "oxidation" to take place. In the other case (air blowing) the same intimacy of contact is insured by treating a much greater bulk of asphalt with a relatively smaller excess of air for a much greater length of time and at a slightly higher temperature. The mechanical equivalency of the two treatments and their resultant similarity of effect upon the physical properties of the asphalt being so clearly indicated, the question reverts to the justification of describing the blowing process as an oxidation.

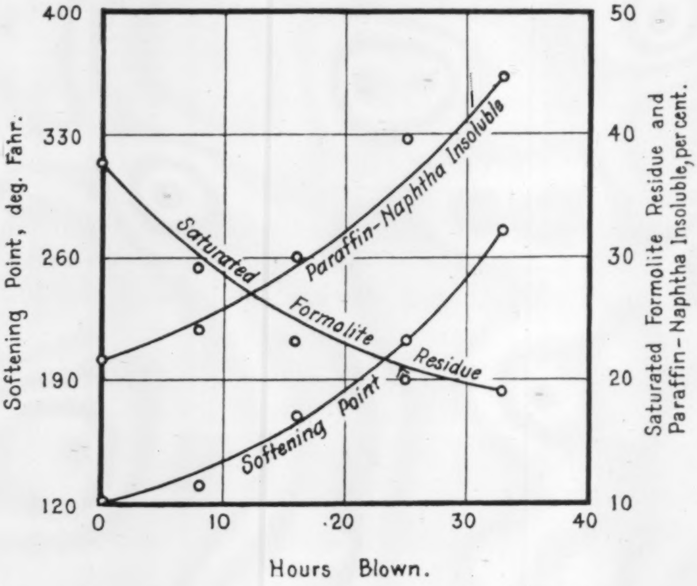

FIG. 1.—Showing Effect of Blowing with Air upon (1) Softening Point; (2) Saturated Formolite Residue; and (3) Oxygenated Bodies Insoluble in 88° Baumé Paraffin Naphtha.

The chemistry involved in this reaction is not any too well understood at best. It seems to be quite generally thought, by men who have studied the subject in this country, that the principal reaction of oxygen on asphalt is a dehydrogenation with an ensuing polymerization of the resultant olefinically unsaturated molecules, all of this taking place without direct combination between the bitumen and oxygen. Published statements, typical of this school of thought, have been made by Brooks and Humphrey,[1] Herbert Abraham,[2] Prévost Hubbard[3] and others. Since the development of the

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[1] Brooks and Humphrey, *Journal Industrial and Engineering Chemistry*, Vol. IX, p. 746 (1917).

[2] Herbert Abraham, "Asphalts and Allied Substances," Second Edition (1920), p. 289.

[3] Prévost Hubbard, "Dust Preventatives and Road Binders."

Mr. Smith.

electron-valence theory, it has long been the practice to consider any reaction involving either the addition of oxygen, the elimination of hydrogen or the increase in valence of an element, an oxidation reaction. From this standpoint alone the authors' use of the term would seem fully justified.

The writer, however, has done some work in this connection which indicates that a direct oxidation takes place during the blowing process. A sample of topped Mexican crude, which had been steam refined to a softening[1] point of 110° F. and a penetration[2] at 77° F. of 225, was blown with air at 450° F. for 33 hours to a softening point of 274° F., samples being taken at regular intervals throughout the run. Detailed analyses were made of the initial, intermediate and final products.

Olefinic unsaturation was found to remain constant by means of Hanus iodine number determinations.[3] Similarly, by means of Nastjulcoff's[4] formolite reaction, the aromatic unsaturation (slightly modified[5]) also remained unchanged. During this reaction, all unsaturated[6] material was either precipitated or eliminated with the oxygenated bodies by means of the sulfuric acid and the residue containing aliphatic and saturated naphtheinic[7] hydrocarbons was found to decrease regularly during blowing. A corresponding increase was observed in the percentage[8] of bitumen insoluble in 88° Baumé par-

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[1] G. & E. Method which averages 10° F. higher than the A.S.T.M. Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36 – 26).

[2] A.S.T.M. Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5 – 25).

[3] *Bulletin No. 107*, revised 1908, Department of Agriculture, Bureau of Chemistry, p. 136. This method was modified by substituting a larger quantity of carbon-tetrachloride for the amount of chloroform specified in order to bring all of the bitumen into solution readily.

[4] Nastjulcoff, *Petroleum*, Vol. 4, pp. 1336 and 1397 (1909).

[5] The method as modified was:

"About 0.25 g. of the sample is weighed into a clean, dry Erlenmeyer flask and dissolved in 35 cc. carbon tetrachloride. This is cooled in an ice bath and 15 cc. concentrated sulfuric acid is run in, drop by drop, with constant agitation in the ice bath. Five cubic centimeters of 40-per-cent formalin is then run in similarly and the mixture allowed to stand 15 minutes. The acid is then diluted, slowly with cooling, with 100 cc. of water and neutralized with concentrated ammonia.

"After fifteen minutes the liquor is decanted through a tared Gooch crucible placed in an adapter in a glass funnel so that the filtrate does not come in contact with the rubber. The filter flask must also be clean. The gummy mass is then washed (in the flask) with water and carbon tetrachloride, the washings being poured through the filter. The heavy emulsion is finally poured into the crucible and heavy suction is applied. The flask is rinsed out with a stream of water from a wash bottle. The filtration completed (this takes from ½ to 3 hours), the black-brown precipitate is stirred or broken with a stirring rod and washed alternately with water and carbon tetrachloride until the washings run clear. It is then sucked as dry as possible and dried in an air bath at 220° F. for one hour. After cooling in a desiccator, the Gooch is weighed and the weight of precipitate is calculated to per cent of original sample.

"The carbon tetrachloride layer is separated in a separatory funnel, washed several times with distilled water, evaporated in a tared beaker almost to dryness and dried in an air bath at 220° F. for one hour. The cooled beaker is weighed and the percent of unattached residue calculated."

[6] Severin, *Mon du Petroe Reum*, 21 (1911).

[7] Herr, *Chemische Zeitung*, Vol. 34, p. 893 (1910).

[8] *Bulletin 1216*, U. S. Department of Agriculture, Bureau of Public Roads, pp. 47–50.

Mr. Smith. affin naphtha, indicating a direct conversion of saturated hydrocarbons to condensed oxygenated bodies.

In Fig. 1 the softening point, the saturated residues from the formolite reaction and the oxygenated paraffin-naphtha-insoluble material are plotted against the blowing time. The marked similarity of all three curves is obvious.

The simplest explanation of the above is in line with the work of Mullerus,[1] Schulz[2] and Kharichkov[3] who oxidized paraffinaceous materials under different conditions to asphalt-like bodies, the process in each case resulting in successive oxidation through alcohols, aldehyde or ketones to complex naphtheinic acids with a loss of side chains in the aldehydric stage of oxidation, indicating that the condensation is similar to the well-known "aldol" type. Surely no other type of reaction would account for the conversion of material, originally saturated, soluble in paraffin naphtha and unaffected by concentrated sulfuric acid, to a different material, still saturated but with the above solubility characteristics reversed.

This work was completed several years ago and no effort has been made since that time to follow it in the literature. Hence, it is possible that additional references have been omitted or that other recently published work has been overlooked.

Mr. Bateman. MR. JOHN H. BATEMAN[4] (*author's closure by letter*).—The determination of penetration at 39° F. instead of 32° F. should have been explained in the paper. We have rather high temperatures in our locality and we found that we could not maintain a temperature of 32° F. without the use of a considerable proportion of salt in the water bath. The standard method for determination of penetration permits the use of brine, but inasmuch as this work was in the nature of a research investigation we did not desire to introduce any complications by the use of salt. This criticism is not a serious one, in the opinion of the authors, as the purpose of the investigation was the determination of the general effect of the mixing process on the physical properties of asphalt rather than the conduct of routine tests to determine the properties of a material which originally had a specific penetration at normal temperature. The variation in penetration between 32° F. and 39° F. of a given sample of asphalt would probably not be any greater than the variation at 32° F. which occurs within the limits of a specific grade (which may vary within a 10-point range at normal temperature). **Mr. Bateman.**

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[1] Mullerus, *Chemische Zeitung*, Vol. 76, p. 1396 (1892).

[2] Schulz, Orig. Com., Eighth International Congress of Applied Chemistry, Vol. 10, p. 273.

[3] Kharichkov, *Chemische Technische Zeitung*, Vol. 28, p. 125 (1907); *Chemische Zeitung*, Vol. 33, p. 1165 (1909); and *Ibid.*, Vol. 34, p. 142 (1910).

[4] Research Professor of Highway Engineering, Louisiana State University, Baton Rouge, La.

The discussion of this paper brought out considerable criticism relative to the use of the term "oxidation" to describe the physical changes which took place in the asphalt as the result of mixing it with hot sand. The term "oxidation" has been widely used to describe the changes brought about in the process of blowing asphalt with air. The process of mixing asphalt with mineral aggregate exposes a tremendous area of hot asphalt, in a thin film, to contact with air and it does not seem unreasonable to suppose that the same kind of effect may thus be obtained as in the case of air blowing. The results of the investigation show that the effect of the mixing process varies only in degree.

The changes effected by the mixing process were certainly not due to sulfur contained in the carbon bisulfide which was used as the solvent. This point has already been covered in the paper; each asphalt used in the investigation was dissolved in carbon bisulfide and recovered by exactly the same process used in recovering the asphalt which had been mixed with mineral aggregate. No significant change in the physical properties of the asphalt took place.

Mr. Miller has raised the question relative to the effect of sulfur in the asphalt in producing the changes which have been described. Until more conclusive information is produced, it is purely a matter of opinion as to whether oxidation in its restricted sense causes the change in physical properties or whether the changes are due to the action of sulfur contained in the asphalt.

The really important feature of this investigation should not be overlooked in this discussion. The big stumbling block in the way of examining asphalt extracted from a paving mixture has been the difficulty in removing all traces of the solvent used in extracting the asphalt from the mixture. This method removes this difficulty with oil asphalt refined from Mexican petroleum.

LOW-TEMPERATURE DUCTILITY OF FILLER-GRADE ASPHALTS

By W. Furber Smith[1]

Synopsis

Samples of filler-grade asphalts meeting the requirements of A.S.T.M. Tentative Specifications for Asphalt Filler for Brick Pavements (D 241 - 26 T) were found to be brittle at low temperatures. Investigation disclosed records of partially blown asphalts being appreciably ductile at 0°C. whereas straight run materials of similar consistency, rarely were. These points were checked by experiment. Analyses of 58 samples of filler grade and paving cement asphalts are reported and results discussed. Substitution of ductility requirement at 0° C. for present ductility requirement at 25° C. and for penetration at 0° C. is recommended.

The purpose of this paper is to call to the attention of the consumers and manufacturers of filler-grade asphalts a characteristic of this material which seems to have been quite generally overlooked and which certainly has not been taken advantage of in the drawing of specifications.

During the last three years a number of samples of filler-grade asphalt were submitted to the testing laboratory of the Oklahoma State Highway Commission with complaints as to their extremely brittle nature at temperatures near freezing. These samples, when tested, met fully the requirements of our specifications which, at that time, conformed closely to the present A.S.T.M. Tentative Specifications for Asphalt Filler for Brick Pavements (Blown Type) (Serial Designation: D 241 - 26 T).[2] After several such samples had been received it was determined to study the low-temperature characteristics of this material with regard to brittleness and ductility, with a view of determining how general this brittleness might be and whether it might be eliminated.

A very brief survey of the literature brought to light a number of instances in which ductility tests were made on various asphalts at 0° C. with results that seemed suggestive. Several discussions touching upon this subject have been given before this Society and, as they seem typical, reference to these alone will be made.

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[1] Assistant Testing Engineer, Oklahoma State Highway Commission, Oklahoma City, Okla.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 866 (1927); also 1926 Book of A.S.T.M. Tentative Standards, p. 559.

In 1906, A. W. Dow[1] quoted results of penetration ranges, ductility determinations at 0 and 25° C. and impact brittleness tests at 0° C., and concluded in part: "the more susceptible to change in temperature, the more ductile and at the same time the more brittle is the material." This conclusion, linking low susceptibility with a general lack of ductility in bituminous materials seems to have been quite generally concurred in for the next several years. As late as 1910 Herbert Abraham[2] concluded similarly: "On the other hand substances endowed with but little ductility will be least affected by changes in temperature."

The following year, however, Abraham[3] published additional data of interest. These results included hardness (consistency), tensile strength and ductility ranges, susceptibility factors and melting points of a number of commercial asphaltic products. His final comment indicates a definite change of attitude: "It is entirely possible to prepare bituminous substances having a low susceptibility factor, and at the same time possessing a relatively great tensile strength and high ductility." Nevertheless he refrained from commenting on one phase of his data which bears directly upon the problem at hand. Out of ten straight-run California and Texas asphalts, no harder in consistency than our present-day filler grades, not one showed a commensurable ductility at 0° C. On the other hand all samples of blown products (including products from the crudes of Texas, Kansas, Oklahoma and the Lima district) having a melting point under 90° C. showed at least a commensurable ductility at freezing temperature. Finally, in 1924, H. B. Pullar[4] in discussion of his own paper before this Society stated: "In connection with the blowing of a Mexican flux a peculiar characteristic has been found, and that is in blowing this flux to an asphalt cement having a penetration of about 50, there is an appreciable ductility at the lower temperatures."

While these published data robbed our proposed investigation of claim to originality, they also opened up a direct avenue of approach. A number of our so-called brittle samples were tested for ductility at 0° C. and each was found to show no ductility under the conditions of the test. It was only with the most extreme care and patience that anything approaching "normal breaks" could be obtained. On the other hand, several samples, concerning which no complaints had been heard, were similarly tested and each showed a ductility under similar conditions of at least 1.5 cm.

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[1] A. W. Dow, "Relations Between Some Physical Properties of Bitumens and Oils," *Proceedings*, Am. Soc. Testing Mats., Vol. VI, p. 497 (1906)

[2] Herbert Abraham, "Improved Instruments for the Physical Testing of Bituminous Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. X, p. 444 (1910).

[3] Herbert Abraham, "Improved Instruments for the Physical Testing of Bituminous Materials, *Proceedings*, Am. Soc. Testing Mats., Vol. XI, p. 673 (1911).

[4] Discussion on Blown Oils by H. B. Pullar, *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 949 (1924).

To obtain information as to the desirability and feasibility of accepting for our use only materials showing this property, request for information was made of a number of large producers with entirely negative results. Hence, ductility determinations at 0° C. were made on all samples until we became convinced that at least two large producers were furnishing a filler grade normally showing ductility at 0° C. A clause was then inserted in our standard specifications requiring a minimum ductility of 1.0 cm. at 0° C. Since that time out of thirty-four shipments to Oklahoma Federal and State Aid Projects only five samples have been obtained which failed to meet this requirement. A summary of the analyses of these samples is shown **in Table I.**

TABLE I.—RESULTS OF TESTS ON FILLER-GRADE ASPHALT.

Sample	Penetration[1] at			Softening Point,[2] deg. Cent.	Ductility[3] at	
	46° C.	25° C.	0° C.		25° C.	0° C.
No. 2527	76	41.5	25	83	4.6	2.5
No. 2587	62.5	30.5	16.5	82	4.7	0.0
No. 2588	63	31.5	20.5	82	4.7	2.2
No. 2805	58	32	21.5	87	3.0	0.5
No. 2822	64	32.5	20.5	80	3.0	2.2
No. 2852	67	34	23	80	3.2	2.3
No. 2875	63	31	24	88	3.0	2.2
No. 2889	53	33	23	80	3.0	0.0
No. 2922	54	29	20	88	2.7	1.7
No. 3286	50	42	29	99	2.2	1.7
No. 3358	63	34	25	81	3.2	2.0
No. 3359	64	33	23	78	3.5	2.0
No. 4114	58.5	31	21	87	4.0	2.2
No. 4139	72	32.5	19	76	3.2	1.7
No. 4150	68	35.5	24	78	3.5	2.3
No. 4302	60	32	20	80	3.3	1.5
No. 4439	61.5	34	25.5	82	3.4	2.1
No. 4608	64	35	26	81	3.7	2.3
No. 4611	69	37	25	80	3.5	2.0
No. 4624	60.5	32	25	79	3.8	2.5
No. 4625	60	29.5	22	79	3.8	0.0
No. 4626	61.5	32	24	79	3.6	2.4
No. 4627	56.5	26.5	22	80	3.6	2.0
No. 4636	67.5	36	27	81	3.1	2.2
No. 4637	65.5	35.5	27.5	81	3.0	1.9
No. 4681	71	38	25	79	3.6	2.2
No. 4709	63	37	23	80	3.0	2.3
No. 4710	68	37	25	80	3.1	2.3
No. 4766	69	37	25	79	4.0	2.5
No. 4791	69	37	19	77.5	...	2.5
No. 4849	66.5	39	27	87	3.5	2.2
No. 5107	59	30	19	75	3.5	2.0
No. 5136	65	32	22	88	5.5	2.0
No. 5146	70	32	19	80	5.0	0.5

[1] A.S.T.M. Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5 - 25).

[2] A.S.T.M. Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36 - 26).

[3] A.S.T.M. Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 - 26 T).

Considerable food for thought is afforded by a brief study of this table. It may be noted that there is a complete gap in the total range of 0° C. ductility values from 1.5 cm. down to 0.5 cm., and it should be added that the two values reported as 0.5 (samples Nos. 2805 and 5146) were hardly true ductilities but rather elasticity values in that a noticeable stretch was observed in each with no observable reduction in area at the center. Practically all of this stretch disappeared immediately after fracture, the fractures in almost all cases occurring at the shoulder of the clip and all being conchoidal in character. On one of these peculiarly behaving samples, upward of 35 briquets were broken at this temperature, only two of which gave normal breaks and both of these showed no commensurable ductility.

It would seem possible that at or near 1.5 cm. there is a critical point below which the material is insufficiently ductile to adjust itself to the strain developed at the shoulders of the briquet clip due to cooling stresses in the relatively large volume of asphalt in the clip. This would account for the remarkably strong tendency of the more brittle samples to fracture at the clip instead of at the point of minimum cross-section. It might be noted here that in Abraham's article[1] referred to above, and also in his well-known text,[2] he reports ductility values under these conditions as low as 0.125 cm. These values were obtained with a radically different type of briquet and it is quite possible that our present mold may be improved upon in this direction. It scarcely seems reasonable that there should be so complete a falling off of ductility values below 1.5 cm. and it is more than possible that refinement of this test may show some limit other than the 1.0 cm. now used by our department, as more desirable.

All of the various brittleness tests that have been applied to asphaltic materials from time to time seem to have fallen into disuse because of their difficulty of reproduction; hence no direct tests of this nature have been applied. The difference in behavior of samples with or without ductility values at 0° C. when subjected to any kind of impact at freezing temperature is so striking as to require very little skill in its observation. In one instance a shipment of drumstock included material from four refinery batches, one of which showed no ductility when tested at freezing. An inspector was able to select every barrel of this batch from the mixed shipment without reference to batch numbers but merely by striking the exposed surface with a hand axe (the temperature was a little below freezing at the time).

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[1] Herbert Abraham, "Improved Instruments for the Physical Testing of Bituminous Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. XI, p. 673 (1911).

[2] Herbert Abraham, "Asphalts and Allied Substances," (1920). Table insert following page 294.

Hence, while there is room for refinement of this test and while our present limit may not be the best, we are at least able to differentiate in the laboratory between material of a more or less brittle nature at low temperatures through the use of a test capable of being reproduced with reasonable accuracy and the value of which is already widely established.

Attention is called to samples Nos. 2587 and 2588 in Table I. They represent two batches shipped from one refinery the same date. There is certainly no difference between the two at any other point to explain their radically different behavior under the ductility test at freezing temperature. One clue as to the possible cause of lack of ductility in sample No. 2587, which fails to show up in the figures given, was observed during testing. Thoroughly experienced operators, using U. S. Bureau of Standards penetrometer needles and thermometers and all using the same penetrometer, encountered great difficulty in obtaining check values for the penetration at 25° C. When several small samples were taken at random from the material, a number of values were obtained at or near 24. Whenever a larger sample (1 to 2 lb.) was melted up carefully close checks were obtained at the value reported. Is it possible that this material had been over-blown, say to a penetration approximating 24, fluxed back to the required consistency and that the incorporation of the flux, though complete enough that the material seemed normally homogeneous when subjected to the regularly applied tests, actually was partly incomplete, say to an extent that would cause a marked separation with an attendant lack of ductility at 0° C.?

Referring again to Table I, samples Nos. 4625 and 4626 furnish another example of two refinery batches part of each of which were included in a single carload shipment. It might be added that they showed respectively ductility values of 8.0 and 11.0 cm. when tested at 46° C. While the first of the two samples shows slightly lower penetrations at all temperatures, still the relationship between their respective ductilities at 46 and at 25° C. would lead one to expect the freezing ductility of the second to be the lower one of the two. Anomalies of this kind make the interpretation of these results difficult indeed.

To get a more definite idea as to the generality of the characteristics observed, samples of 50 to 60, 40 to 50 and 30 to 40 penetration paving cements and of 30 to 40 penetration filler-grade asphalts were obtained from six large refineries whose products are widely used throughout the country. Penetration and ductility ranges and softening point determinations were run on each. The results of these tests are given in Table II. From a brief study of this table the following points should be noted. Of six straight-run 50 to 60 penetration asphalts, four exhibited ductility at 0° C. Of six 40 to 50 paving cements, only one showed this property and not one of the 30 to 40 grade exhibited any tendencies in this direction. Of the six partially blown or filler-grade asphalts, all but one gave ductility values of 1.5 cm. or greater at freezing temperature, and as this paper is written we have at hand a letter from the refinery whose sample of filler-grade asphalt failed in this respect, advising that tests made in their laboratory on another refinery batch gave a value of 1.5 cm.

TABLE II.—COMPARISON OF FILLER GRADE WITH PAVING CEMENT ASPHALTS FROM SAME SOURCES.

Refinery	Grade, penetration	Type	Sample Number	Penetration[1] at			Softening Point,[2] deg. Cent.	Ductility[3] at		
				46° C.	25° C.	0° C.		46° C.	25° C.	0° C.
A	50 to 60	Paving Cement	4796	211	49	17	54	100+	100+	3.0
	40 to 50	Paving Cement	4795	175	44	18	58	100+	100+	0.0
	30 to 40	Paving Cement	4794	115	31	12	62	100+	100+	0.0
	30 to 40	Filler Grade	5145	79	32	18	74	38	12	0.5
B	50 to 60	Paving Cement	4667	...	56	20	58	100+	78	3.3
	40 to 50	Paving Cement	4738	158	45	20	60	100+	43	2.7
	30 to 40	Paving Cement	4820	97	30	15	64.5	100+	19	0.0
	30 to 40	Filler Grade	4626	61.5	32	24	79	11	3.6	2.4
C	50 to 60	Paving Cement	4800	210	57	21	54.5	100+	100+	2.5
	40 to 50	Paving Cement	3988	178	43	17	58	100+	100+	0.0
	30 to 40	Paving Cement	4799	138	35	11	58	100+	72	0.0
	30 to 40	Filler Grade	4801	83	35	19	83	26	9	2.0
D	50 to 60	Paving Cement	4784	204	48	15	56	100+	100+	0.0
	40 to 50	Paving Cement	4783	168	42	12	57.5	100+	100+	0.0
	30 to 40	Paving Cement	4782	135	33	11	62	100+	100+	0.0
	30 to 40	Filler Grade	4785	62	34	20	84	11	5	1.5
E	50 to 60	Paving Cement	5116	226	58	19	53	100+	100+	0.0
	40 to 50	Paving Cement	5117	192	47	17	56	100+	53	0.0
	30 to 40	Paving Cement	5118	162	35	13	58	100+	55	0.0
	30 to 40	Filler Grade	5119	64	38	24	81	8	3.5	2.0
F	50 to 60	Paving Cement	5167	241	58	18	55	100+	100+	[illegible]
	40 to 50	Paving Cement	5166	174	44	14	59	100+	100+	0.0
	30 to 40	Paving Cement	5165	...	..	..	..	100+	100+	0.0
	30 to 40	Filler Grade	5135	72	35	23	73	26	8.5	2.0

[1] A.S.T.M. Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5 - 25).

[2] A.S.T.M. Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36 - 26).

[3] A.S.T.M. Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 - 26 T).

Looking again at the results on the products of refineries D and E, the presence of freezing temperature ductility in the blown product in spite of its complete absence in softer grades of straight-run material is even more significant.

It has long been recognized that the low susceptibility factor and the high softening point of the partially blown asphalt make it admirably adapted for filler work. These properties have been demanded by consumers through specifications for a number of years. It is believed that the data presented, though by no means voluminous, is adequate to show the feasibility of taking advantage of this other property of this type of asphalt.

The desirability of utilizing this property of low-temperature ductility is obvious if consideration be given to the kind of service demanded of modern filler asphalts. Not distended over the surfaces of a mineral aggregate and protected by the very density of the paving mix but exposed to the abuse of traffic in sections from one-eighth inch or less (in brick filler work) to an inch or more (in poured type expansion joints) in thickness, it is expected to withstand repeated blows and considerable movement (due to contraction and expansion of the slab itself) without parting company either with itself or with its surroundings in the coldest weather. It scarcely seems possible that a set of conditions could be formulated which would create a greater demand for ductility in an asphaltic product. Even at the present time we are setting a lower limit for consistency (penetration) at 0° C., thus indirectly requesting a minimum of ductility. Would it not be highly logical to require a freezing temperature ductility test, eliminating entirely the present ductility requirement at normal temperature and, if desired, the freezing temperature penetration?

In summing up, it is believed that the following conclusions may be drawn legitimately from the evidence presented:

1. Partially blown asphalts of the type usually designated as "filler grade" are normally ductile at 0° C.
2. This property is an additional (to those already widely recognized) and characteristic difference between this type and straight-run asphalts.
3. This property is highly desirable in a filler-grade asphalt and its requirement (directly specified) should be substituted for present normal temperature ductility requirements and possibly for 0° C. penetration requirements.
4. The type of ductility briquet mold now in use should be studied with regard to strains set up in samples cooled for this test.

Acknowledgment.—Acknowledgment of indebtedness is made to Frank Herrmann, Testing Engineer for the Oklahoma State Highway Commission for permission to publish the data given, to C. W. Barbour of this laboratory who made most of the special tests involved, and to the refineries that furnished the special samples used.

DISCUSSION

Mr. Pullar.

MR. H. B. PULLAR.[1]—I was very much interested in reading Mr. Smith's paper, in view of the fact that it confirms, to a considerable extent, the contentions that I have made for some time as to the value of ductility. If it is true, as the advocates of ductility contend, that it indicates the mobility and binding properties of an asphalt, then it has always seemed to me that a ductility taken at a low temperature would be of more value than the ductility taken at a normal temperature. However, I doubt very much whether the present method of taking ductility tests is such that the conclusions which the author has drawn would be warranted. I do not think that the test as it is now standardized is capable of giving check results, particularly on material having low ductility. The personal equation, the method of handling briquets at low temperatures, and the difficulty in maintaining a constant low temperature makes it difficult to check results.

My own experience confirms the statement of the author that most blown asphalts normally show a ductility at 0° C. and that they invariably show a much less susceptibility to changes in temperature.

I was particularly interested in the results which the author obtained on the paving cement samples. I do not know what "refinery B material" refers to nor the reason for the ductility of the three samples at 0° C. It might have been due to the crude oil from which the material was produced or to the process by which the material was made, but it does confirm my previous statements that it is possible to get a paving cement with a high ductility at normal temperatures and an appreciable ductility at 0° C. I believe that the time is coming when there will be a large demand for an asphalt cement having a ductility at low temperatures and I think this paper indicates a tendency on the part of at least the consuming trade to give more consideration to this desirable characteristic. I have found from experience that treating Mexican asphalt with air makes it possible to get a paving cement between 50 and 60 penetration that has a ductility of over 60 cm. at 25° C. and a ductility as high as 12 cm. at 0° C.

I also had some very interesting results from blending high ductile materials with blown mid-continent oils. In one case I found it possible to get a paving cement having a ductility at 25° C. of 4.5

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[1] Secretary and Manager, Heavy Oil Sales, James B. Berry's Sons Co., Chicago, Ill.

Mr. Pullar. cm. and a ductility of 3.75 cm. at 5° C. This paving cement has a very low susceptibility factor, indicated by the following penetrations:

Penetration at 25° C. (100 g., 5 sec.)	30
Penetration at 0° C. (200 g., 1 min.)	13
Penetration at 46° C. (50 g., 5 sec.)	50

On this same blend, with the paving cement at penetration of 60 instead of 30, the ductility at 25° C. was between 50 and 60 cm. and the ductility at 5° C. was 9 cm. with a low susceptibility, as shown by the following penetrations:

Penetration at 25° C. (100 g., 5 sec.)	59
Penetration at 0° C. (200 g., 1 min.)	31
Penetration at 46° C. (50 g., 5 sec.)	130

While not a recognized standard test, the cementing value taken on the Kirschbaum instrument showed a considerable increase in the cementing value of this product over the higher ductile cements of the same penetration, which in my opinion was further confirmation of the desirability of a ductility at low temperatures.

I do not agree with the author of the paper in his explanation for getting no ductility at 0° C. temperatures on some of the samples. I presume in referring to "over-blown" he meant that the product was carried beyond the desired penetration or the desired melting point, and then fluxed back with either a softer asphalt or a fluxing material; but there has been considerable misunderstanding by a great many engineers and chemists as to the "over-blowing" of asphalt and the injury to blown products by blowing at high temperatures. From considerable experience and from the results of many tests made by blowing at high temperatures, I am of the opinion that there is no such thing as "over-blowing" or injuring the product by action of the air. I have found that there is much more probable chance of blowing up your plant and having a fire than there is of having the resulting product "over-blown." Asphalts that have been blown at temperatures as high as 550° F., so far as any determinations that I have seen or made, show no difference in characteristics from material blown from 450 to 500° F. If one attempts to blow oil at temperatures above 550° F., one is very apt to have spontaneous combustion.

While there is probably a critical temperature in blowing, most of the trouble that I have experienced with blown asphalts has been due principally to the crude materials from which they have been produced rather than from the blowing operation. In blowing oil there are three correlative factors to be taken into consideration:

Mr. Pullar.

temperature, volume and distribution of air, and the manner in which the fumes are exhausted. This last factor is of considerable importance as one will get quite different results by exhausting the fumes as fast as they are generated than if one permitted these fumes to condense and fall back into the blowing oil.

I feel that the author has, by this paper, again brought to the attention of engineers and chemists a most desirable characteristic of blown asphalts and a characteristic that will be given more consideration in the future.

Mr. Dow.

MR. A. W. DOW.[1]—Mr. Smith's paper is of great value and it brings out some very interesting points, but I do object to his not stating his results in ductility in accordance with the instructions of our tentative method. I have not a copy of these instructions with me, but in his results that he calls zero ductility he really has not tested for ductility but only brittleness. If a more carefully constructed apparatus had been used, the samples which broke in the clips and to which he has assigned a ductility of zero might have pulled to a considerable length. I have done a great deal of work on the ductility of brittle materials and have even succeeded in obtaining the ductility of California asphalt at 0° C. This is one of the most brittle asphalts on the market, far more so than many that Mr. Smith has tested and has marked as zero ductility at 0° C. The determination on the ductility of California asphalt was obtained on a machine far better and more carefully constructed than any of the ductility machines now on the market. If ductility tests are to be made on very brittle materials better screws and gearing should be used in the ductility machine. The slightest jar to a brittle sample while in tension will cause it to break due to brittleness.

Mr. Ross.

MR. E. S. ROSS.[2]—It has been my experience with the ductility test at 0° C. that the ductility machines are much more temperamental than the operators. I have tested ductility on a number of machines, and find some that start off with a jerk, and materials that have a low ductility are recorded as having zero ductility because the sample snaps off, whereas if that same sample were run on another ductility machine which starts without that jerky motion, one would get possibly 1 or 1.5 cm. ductility. I wonder if consideration has been given to the ductility machine in accounting for the fact that very few ductility values have been obtained below 1.5 cm.?

Mr. Miller.

MR. J. S. MILLER.[3]—It is unfortunate to introduce all these different temperatures. Why go to 0° C. when 5° C. is so much more

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[1] Dow and Smith, Paving and Chemical Engineers, New York City.

[2] Senior Industrial Fellow, Mellon Institute of Industrial Research, Pittsburgh, Pa.

[3] Director, Technical Bureau, The Barber Asphalt Co., Maurer, N. J.

Mr. Miller. easily obtained and maintained? If we take the flat bed machine, it is very difficult to maintain 0° C. temperature. It is much easier to do it at 5° C. and I think in considering these grades of asphalt that tests at 5° C. will give just as much information as those at 0° C.

Mr. Dow. MR. DOW.—It seems to me that this problem of Mr. Smith shows the necessity of developing a brittleness test. A brittleness test is really valuable in some lines of asphalt investigations and while there has been some work done on it we have not as yet devised an apparatus that will give us concordant results. The New York State Highway Department has a brittleness test which was developed by them to determine the same property that Mr. Smith is endeavoring to obtain by taking the ductility at 0° C. New York State found that some materials irrespective of their ductility at 25° C. were far more friable and brittle at low temperatures than other materials showing the same ductility. Some of these were so brittle that when used in pavements they readily ground up under traffic in cold weather. I am not sufficiently acquainted with the work of New York State to say whether concordant results are obtained by its method, but I fully believe that an earnest endeavor should be made to devise an apparatus for the determination of the brittleness of bitumen.

Mr. Weiss. MR. J. M. WEISS.[1]—I know that at the time I was with The Barrett Co., we developed a machine, a description of which was presented before this Society,[2] with which we could obtain very concordant breaking point results. Those tests checked always within 1° C., with several operators and with different materials.

Mr. Reeve. MR. C. S. REEVE.[3]—That is correct, I think. The principle of the impact brittleness test, such as used by New York State at times, and the prism test, which Mr. Dow himself developed when used in the asphalt blocks, was the rupture of a standard form of test piece by impact. The type of machine we developed and described before this Society three years ago[2] was really a breaking point test—the determination of the maximum temperature at which the material would break when bent under standard conditions. That machine does, as Mr. Weiss states, give results quite readily within 1° C. In other words, the breaking point over a number of repeated determinations on a given material will not vary more than 1° C. The test was

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[1] President, Weiss and Downs, Inc., New York City.

[2] C. S. Reeve and F. W. Yeager, "Machine for the Determination of the Pliability of Prepared Roofing and the Breaking Point of Bitumen," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 385 (1925).

[3] Chief Chemist, Development Department, The Barrett Co., Leonia, N. J.

Mr. Reeve. standardized for our own purposes only, and is subject to further standardization for other purposes. That is the best method I know of for making a breaking point determination that is reproducible between different operators in different laboratories, and it might very well be applied to a further study of the filler proposition.

Mr. Ross. MR. ROSS.—Messrs. Bateman and Delp's paper[1] refers to the penetration test at 39° F. I believe that Mr. Pullar, in one of his analyses, referred to the same test at 5° C. (41° F.), and Mr. Miller is of the opinion that we ought to run the test at that temperature. I wonder if there is sufficient reason or desire for our looking into this matter? In spite of the fact that we now specify 0° C., is there sufficient reason for our consideration in bringing about a revision? It does seem, from what was said this morning, that there are more desirable temperatures than 0° C. for the determination of penetration at low temperature.

Mr. Milburn. MR. H. M. MILBURN.[2]—I should like to ask if the percentages of bitumen (soluble in carbon bisulfide), percentages of organic matter insoluble, and specific gravities were determined on the samples reported in Table I, and, if so, if a correlation of the results of these tests with the results of the ductility tests at 0° C. gives any significant data.

Mr. Smith. MR. W. FURBER SMITH.[3]—May I refer briefly to the several comments that have been made? In Mr. Pullar's discussion, the ambiguity of my use of the term "over-blown" was brought out. It should be explained that by over-blowing was meant merely the carrying of the normal blowing process a little too far, resulting in a product harder than had been desired. Reference to the possible use of too much air and too little steam or the use of objectionably high temperatures was not intended. It was to the possible incomplete incorporation of a softening medium that I intended to refer as a possible source of difficulty, and this reference was made more for the purpose of eliciting discussion than from very strong conviction in that direction.

Mr. Dow's criticism of my method of reporting ductility values as 0° C. is justified. I was at loss for a better method of expression in a tabulation. You will observe that in the text it is stated that the "material showed no ductility under conditions of the test." With regard to the feasibility of checking these results with the equipment in use, the machine used was a practically new Howard

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[1] John H. Bateman and Charles Delp, "The Recovery and Examination of the Asphalt in Asphaltic Paving Mixtures," see p. 465.

[2] Chemist, U. S. Bureau of Public Roads, Washington, D. C.

[3] Assistant Testing Engineer, Oklahoma State Highway Commission, Oklahoma City, Okla.

Mr. Smith. & Morse machine. The molds used were from three different manufacturers and one set was discarded because of its tendency to give fewer normal breaks than the others. Except with the "elastic" samples referred to, but little difficulty was experienced in obtaining checks.

The questionable suitability of our present mold is also referred to in the paper. During the progress of the investigation, an attempt was made to obtain molds of the type developed by Herbert Abraham for his consistometer which was described before this Society some years ago.[1] Failing in this effort, a mold was made up with a minimum cross-section of 1 cm. square and having a horizontal section similar to a section through the axis of the Abraham mold. With this mold, normal breaks were, of course, obtained each time and checking results was quite easy. Samples which were reported in the tabulation as having zero ductility again failed to register. Samples reported with ductilities of 1.5 cm. or greater gave values considerably smaller but quite readily checkable.

With regard to the relative merits of 0° C. as compared with 5° C. for the temperature of the test, it might be stated that 0° C. was selected for this work for its ease of maintenance for prolonged time intervals. No trouble whatever was experienced in this connection so long as ample quantities of finely crushed ice were used. It was found necessary to bring the entire volume of liquid in the ductility machine to temperature; packing the third nearest the breaking end seemed adequate to maintain a temperature within 0 to 0.5° C.

Replying to Mr. Milburn's questions the data as to solubility in carbon bisulfide, carbon tetrachloride, etc., were not complete enough to justify publication. In the few cases where complete analyses were available no difference could be observed between samples (from the same source) which were normally ductile or less ductile at freezing.

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[1] Herbert Abraham, "Improved Instruments for the Physical Testing of Bituminous Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. IX, p. 568 (1909); see also Vol. X, p. 444 (1910); see also Vol. XI, p. 673 (1911).

A STUDY OF THE INFLUENCE OF MOISTURE ON RUBBER TESTING

By Fred S. Conover[1] and Harlan A. Depew[2]

Synopsis

The object of this investigation was to determine the effect of moisture on cured and uncured rubber stocks and, if this effect was pronounced, to determine whether or not the testing accuracy could be increased by the control of relative humidity. If accuracy could be increased, it was also desirable to find the best relative humidity at which to carry out the work.

The method followed was to expose uncured slabs to various relative humidities for increasing lengths of time, then to cure them and make tests for tensile strength, stress at 300 and 450 per cent elongation, slope and ultimate elongation. Cured samples were then exposed and tested in the same way.

The results show, in general, that moisture plays a very large part in the physical properties of rubber goods, whether it is absorbed before or after curing, and that the amount of change is roughly proportional to the amount of water absorbed.

The conclusions are that it is very desirable to control relative humidity, and that zero per cent relative humidity is the preferred condition, because at a low humidity the changes which take place in the moisture content of the stocks are reduced to a minimum and absolute dryness is the easiest condition to maintain.

Introduction

A great deal of the work of our laboratory consists in the testing of zinc oxides for rubber purposes. This is divided into two parts: the control grading of shipments and the evaluation of experimental zinc oxides. The testing of zinc stocks with their well-known knotting properties is unusually difficult and we realized early in our work that it was necessary to study our testing methods and improve upon them.

As a result of this study and the subsequent introduction of a number of innovations, we increased the accuracy of our testing very appreciably. Among the innovations were the installation of laboratory roll thermometers which enabled our laboratory operators to mill the rubber at constant temperature, and the adoption of a constant-temperature testing room.

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[1] Research Division, New Jersey Zinc Co., Palmerton, Pa.

[2] Research Division, New Jersey Zinc Co., Palmerton, Pa.

In order to obtain the greatest value from our testing laboratory we feel that it is necessary still further to increase our testing accuracy. We have noticed that a recure of a stock that has lain in the laboratories for a couple of weeks often will not check earlier tests. We have also observed that a "laboratory control sample" of a definite formula tests differently from season to season.

It has been suggested, from analogy to paint testing,[1] that variability in moisture content might play an important part in explaining these discrepancies. There were no published rubber tests upon which to base this conclusion until Stringfield's paper[2] was presented, in which he gave data showing that relative humidity was a real factor. This paper, however, did not make any recommendations for laboratory control and the question was left unanswered as to whether it was necessary to apply humidity control during milling, whether it was necessary to apply a control to the crude rubber before milling, and what humidity control should be adopted.

We were sufficiently impressed by the importance of this report to start a testing program to check Stringfield's conclusions, and to standardize on a humidity control, if we found it desirable.

In this program we assumed that the testing variation produced by different conditions of humidity depended on the variations of moisture content, and that conditions of laboratory control should be chosen that would keep the moisture content as nearly constant as possible.

PROCEDURE DATA AND DISCUSSION

In order to carry out our experimental work it was necessary to have a continuous stream of dry air passing through one storage cabinet and a corresponding stream of moist air passing through another. Dehydration by silica gel was the means decided upon for securing the dry air. For the moist stream, air was bubbled through three bottles containing water and glass wool.

Three widely divergent mixes were chosen as being representative stocks. The first was of a highly hygroscopic nature, containing latex sprayed crude rubber, hexamethylene-tetramine and glue. The second contained less hygroscopic material, pale crepe, XX Red zinc oxide and Diphenylguanidine. The third was a reclaim compound containing litharge as the accelerator. These compounds are:

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[1] H. A. Nelson and G. W. Rundle, "Further Studies of the Physical Properties of Drying-Oil, Paint and Varnish Films," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 356 (1923).

[2] R. B. Stringfield, "The Effect of Humidity on Rubber Testing," *Industrial and Engineering Chemistry*, Vol. 17, pp. 833–835, August, 1925.

COMPOUND A		COMPOUND B		COMPOUND C	
Latex Sprayed Rubber	920	Pale Crepe	920	Whole Auto Tire Reclaim	742[a]
Sulfur	55	Sulfur	55	Pale Crepe	460
Glue	67½	D.P.G.	4½	Sulfur	74
Hexa	9¼	XX Red Zinc Oxide	1035	Litharge	185
XX Red Zinc Oxide	1035			XX Red Zinc Oxide	1035

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[a] Contains 460 parts (500 volumes) of rubber hydrocarbon.

Variations in Moisture Content:

Each of these batches was mixed on a 16 by 42-in. mill and allowed to cool. Samples of 500 g. were then taken and, after an interval of about a week, were placed in the wet and dry cabinets for 10 and 20-day periods, after which they were weighed. Table I shows the amounts of moisture taken up or lost by these samples. Since the stocks were allowed to lie around to pick up moisture we can observe change in both directions.

TABLE I.—CHANGE IN MOISTURE CONTENT OF UNCURED STOCKS WHEN EXPOSED TO 0 PER CENT AND 100 PER CENT RELATIVE HUMIDITIES.

Values are expressed in grams change in weight of 500-g. samples

	EXPOSURE PERIOD			
	10 DAYS		20 DAYS	
	WET	DRY	WET	DRY
Compound A	+1.7	−2.6	+2.3	−2.8
Compound B	+0.6	−1.1	+0.6	−1.1
Compound C	+0.8	−1.9	+1.3	−2.0

The samples kept dry show a gradual loss of their original moisture content as the exposure periods lengthen. At the end of the 20-day period they contained practically the same amount of moisture which they had when they were cut off the mill. The hygroscopic compound A, since it had the highest initial moisture content, lost the greatest amount of moisture. Compound B had the lowest initial moisture content and lost less weight than compound A. The reclaim mix, compound C, showed a much faster rate of change in weight than compound B and was nearly as hygroscopic as compound A that was chosen from especially hygroscopic materials. This high moisture absorption of a reclaim stock is, incidentally, a source of trouble in manufacturing. It is our opinion that more trouble, due to blistering, is caused by failure to dry reclaim mixes thoroughly than from all other reasons.

The samples kept in the wet box show steady increases in weight as the exposure periods lengthen with the exception of compound B which is practically at equilibrium at the end of ten days.[1] The others have not come to equilibrium and show no indications of doing so, whereas the dry samples are practically thoroughly dehydrated.

These same samples, after being weighed, were milled under wet and dry conditions. Table II shows the moisture gained or lost under this treatment. An examination of this table shows that samples which were conditioned in the wet cabinet lost weight upon milling, whether this milling was done wet or dry. Samples which were kept dry and milled wet gained, on the average, small amounts in weight. Those which were kept dry and milled dry lost very slight amounts.

TABLE II.—CHANGE IN MOISTURE CONTENT CAUSED BY MILLING[a] UNDER WET AND DRY CONDITIONS.

Values are expressed in grams change in weight of 500-g. samples.

		EXPOSURE PERIOD BEFORE MILLING			
		10 DAYS DRY	20 DAYS DRY	10 DAYS WET	20 DAYS WET
Hexa.	A Milled Wet.	+0.50	+0.64	−0.95	−1.3
	A Milled Dry.	−0.08	−0.05	−0.14	−0.86
D.P.G. . . .	B Milled Wet.	+0.17	−0.06	. . . . . .	−0.48
	B Milled Dry.	0.00	−0.02	−0.80	−0.45
Reclaim. . .	C Milled Wet.	+0.22	0.00	−0.55	−0.18
	C Milled Dry.	0.00	0.00	−0.55	−0.63

[a] Milling carried out at 70° C.

During the wet milling, steam was shot directly onto the rubber. It was so wet that drops of water were continually forming on the jet and dropping into the rolls. In spite of this the amounts of weight gained were astonishingly small in comparison with the amounts gained during the exposure periods, and at the same time are very large in comparison with the amounts gained under ordinary conditions. The temperature at which the rolls are working and therefore of the air next to the rolls is probably great enough to keep the relative humidity of the air which comes into contact with the rubber at a very low percentage. To express this thought quantitatively, air at

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[1] C. R. Boggs and J. T. Blake, in the *Journal of Industrial and Chemical Engineering* for March, 1926, Volume 18, No. 3, have published data on the rates of absorption of water by uncured samples of different rubbers. They found wide variations, depending on temperature, osmotic pressure, surface tension, and the type and concentration of the coagulant. Reclaimed rubber was found to have a very high rate of increase. Most of their work was done by actually immersing the samples in distilled water whereas the work here has been done at 100 per cent relative humidity. However, it is probable that rates of absorption are not much different at 100 per cent relative humidity from what they are at actual immersion for the same length of time. (See also the *India Rubber Journal*, Vol. 73, February 12, 19 and March 5, 1927, article by Williams and Kemp, and *India Rubber Journal* for March 1927, article by J. D. Fry.)

100 per cent relative humidity and atmospheric temperature will only have a relative humidity of about 7 to 12 per cent when heated to 70° C. in contact with the rubber on the mill; whereas, when water actually touches rubber the relative humidity is 100 per cent at that temperature. It is very probable, therefore, that all milling is comparatively dry, regardless of the relative humidity of the atmosphere at the time.

Assuming this to be a fact, our argument for making dryness the standard condition for testing is further substantiated. The rubber leaves the mill fairly dry and it is comparatively easy to keep a stock dry when it is already in that condition.

TABLE III.—THE EFFECT ON TENSILE PROPERTIES CAUSED BY DIFFERENT HUMIDITIES BEFORE AND DURING MILLING.

Stocks were all kept at the same (laboratory, about 50 per cent) relative humidity between curing and testing (24 hours) Each value average of 5 tests.

	Condition Before Milling	Condition During Milling	Condition Between Milling and Curing (24 hours)	Tensile Strength, lb. per sq. in.		Elongation, per cent		Stress at 300 per cent Elongation, lb. per sq. in.		Stress at 450 per cent Elongation, lb. per sq. in.		Slope,[b] lb. per sq. in.	
				10 Days[a]	20 Days[a]	10 Days	20 Days	10 Days	20 Days	10 Days	20 Days	10 Days	20 Days
Compound A	Wet	Wet	Wet	3025	2790	530	500	1340	1300	2535	2485	1195	1185
	Wet	Dry	Dry	3125	2860	540	515	1290	1275	2470	2435	1080	1160
	Dry	Wet	Wet	3165	3085	545	530	1305	1315	2460	2505	1155	1190
	Dry	Dry	Dry	3230	3140	540	550	1335	1290	2560	2420	1225	1130
Compound B	Wet	Wet	Wet	2805	2885	625	620	575	600	1350	1455	755	855
	Wet	Dry	Dry	2985	3000	620	625	645	655	1500	1465	855	810
	Dry	Wet	Wet	3195	3115	615	615	795	780	1725	1740	930	960
	Dry	Dry	Dry	3225	3010	605	595	845	835	1830	1750	985	915
Compound C	Wet	Wet	Wet	1470	1420	420	400	1035	1075	....	....	....	....
	Wet	Dry	Dry	1525	1495	410	400	1100	1125	....	....	....	....
	Dry	Wet	Wet	1505	1470	400	385	1140	1165	....	....	....	....
	Dry	Dry	Dry	1550	1490	410	390	1255	1155	....	....	....	....

[a] Exposure period before milling.

[b] Slope is the difference between stresses in pounds per square inch at 450-per-cent and 300-per-cent elongation.

Effect of Moisture Conditions on Tensile Properties:

These samples after wet and dry milling were cured and tested in accordance with the A.S.T.M. Standard Methods of Testing Rubber Products (D 15 – 24). Table III shows the effects of these treatments on tensile strength and stiffness.

All results given represent the averages of five tests. Table III shows that samples which were kept dry have a higher tensile strength than corresponding samples which were kept wet. It is further evident that wet milling has had a slightly deleterious effect on tensile strength, but in view of the fact that the rubber was in actual contact with water during milling this effect was exaggerated and it would probably be entirely negligible even in the case of saturated air. The elongations are practically the same for all corresponding mixes. All samples show a steady decrease in tensile strength as the conditioning periods are completed. That is, the 20-day samples are inferior to the 10-day samples, and these in turn are inferior to the original. This is true of both wet and dry samples.

It has long been recognized that stocks should stand for some time after milling, perhaps from 24 hours to a week. These findings indicate that there is, at the same time, an aging effect that works in the opposite direction. Possibly the sulfur and accelerator have a deteriorating effect on goods if allowed to remain uncured for too long a time.

TABLE IV.—THE EFFECT ON TENSILE PROPERTIES CAUSED BY 0 PER CENT AND 100 PER CENT RELATIVE HUMIDITIES WITHOUT REMILLING.

	Exposure Before Curing	Tensile Strength, lb. per sq. in.			Elongation, per cent			Stress at 300 per cent Elongation, lb. per sq. in.			Stress at 450 per cent Elongation, lb. per sq. in.			Slope, lb. per sq. in.		
		10 Days[a]	20 Days[a]	30 Days[a]	10 Days	20 Days	30 Days	10 Days	20 Days	30 Days	10 Days	20 Days	30 Days	10 Days	20 Days	30 Days
Compound A	Wet	2810	2630	2720	550	615	560	1090	760	570	2315	1615	1915	1045	890	1345
	Dry	3105	3090	3040	535	565	545	1215	950	1190	2425	1865	2300	1210	915	1110
Compound B	Wet	3180	2975	2900	625	630	625	698	645	565	1485	1425	1275	787	780	710
	Dry	3330	3180	3180	605	615	600	840	760	750	1825	1650	1680	985	890	930
Compound C	Wet	1315	1220	Blistered	385	380	Blistered	1050	1005	Blistered	....	....	....	....	....	....
	Dry	1410	1420	1470	380	400	400	1110	1080	1090	....	....	....	....	....	....

[a] Exposure period before curing.

Since samples kept dry and milled wet are higher in tensile strength and stiffness than corresponding samples kept wet and milled dry, it follows that exposure to different atmospheric humidities is a more important factor than wet and dry milling.

Stringfield found that if a stock were accelerated with Hexa, the slope would increase as the relative humidity before curing increased. The slope figures for the compound A given in Table III show this to be true, but only after the stock has been subjected to 100 per cent relative humidity for 20 days. He defines slope as the difference in stresses at 500-per-cent and 700-per-cent elongations and is dealing with almost pure gum stocks. We were forced to define slope as the difference in stresses at 300-per-cent and 450-per-cent elongations because our samples broke before reaching 700-per-cent elongation, and we are using rather heavily loaded mixes. Therefore, we believe that both results are entirely in line, one with the other.

In stocks accelerated with D.P.G., the factors which have obscured the conclusions for Hexa stocks seem to have made no difference whatever. For every increase in humidity there is a corresponding decrease in the slope figure, which is in exact accordance with Stringfield's work.

Since the humidity conditioning periods are found to be of more relative importance than the milling, samples of compounds A, B and C were placed in the dry and wet cabinets for 10, 20, and 30-day periods and then cured and tested without being milled again. The results are given in Table IV. The samples of compound C blistered so badly when exposed to a 100 per cent relative humidity for 30 days that accurate tests were impossible.

TABLE V.—EFFECT ON TENSILE PROPERTIES OF EXPOSURE OF UNCURED SAMPLES TO VARIOUS RELATIVE HUMIDITIES FOR SHORT LENGTHS OF TIME.

	Exposure Period Before Curing, Hours[a]	Compound A				Compound B				Compound C			
		Relative Humidity, per cent											
		0	50	80	100	0	50	80	100	0	50	80	100
Tensile Strength, lb. per sq. in..	24	3060	3010	2980	3010	3280	2880	3200	3260	1520	1470	1500	1440
	48	3060	2900	2900	2980	3340	2945	2980	3140	1550	1470	1390	1440
	72	2840	2995	2980	2900	3080	3020	2940	2840	1590	1530	1540	1440
	144	3040	3020	2950	2920	3160	3145	3040	2930	1345	1600	1485	1560
Elongation, per cent..........	24	520	515	520	545	595	580	605	630	400	390	395	385
	48	535	540	520	545	610	595	600	610	395	405	390	380
	72	505	550	520	530	600	600	605	620	410	415	405	425
	144	555	565	560	565	...	615	630	640	415	445	425	430
Stress at 300 per cent Elongation, lb. per sq. in	24	1240	1240	1240	1240	835	780	770	805	1150	1090	1110	1150
	48	1180	1170	1130	1240	705	690	715	675	1060	1040	1080	1025
	72	1120	1150	1170	1140	740	715	705	555	1110	1010	1020	1010
	144	1210	1200	1170	1075	695	580	640	580	1000	1040	1035	1060
Stress at 450 per cent Elongation, lb. per sq. in	24	2515	2400	2400	2400	1890	1780	1730	1800	....	....	....	....
	48	2330	2340	2340	2360	1630	1640	1620	1570	....	....	....	....
	72	2350	2270	2370	2250	1680	1660	1555	1330	....	....	....	....
	144	2310	2290	2170	2110	1580	1585	1470	1320	....	....	....	....
Slope, lb. per sq. in.........	24	1275	1160	1160	1160	055	1000	960	995	....	....	....	....
	48	1150	1170	1210	1120	925	950	905	895	....	....	....	....
	72	1230	1120	1200	1110	930	945	850	780	....	....	....	....
	144	1100	1090	1000	1035	885	905	830	740	....	....	....	....

[a] Exposure period before curing.

These data substantiate those given in Table III. The gradual lowering of the tensile strength of stocks exposed to high humidity is shown just as before, as well as the peculiar reversal of the slope figure for the Hexa mix which was exposed for 30 days to 100 per cent relative humidity.

Effect of Relatively Short Periods of Exposure:

All of our results so far have shown that exposure to wet and dry conditions makes a difference in the quality of the stocks which have undergone such treatment. However, the work was carried out over periods of time and under conditions which are rarely, if ever, encountered in a laboratory. Simply because differences in tensile strength of 200 to 500 lb. per sq. in were found in corresponding samples exposed for lengthy periods at 0 per cent and 100 per cent relative humidity, it does not necessarily follow that the amounts of change will be the same, or even proportional, when the work is done over periods of 24, 48, 72 and 144 hours and at relative humidities which vary by, perhaps, only 30 per cent. In order to find the differences which would result from relatively short periods of exposure and slightly varying humidities, uncured samples of compounds A, B and C were exposed for 24, 48, 72 and 144-hour periods at 0, 50, 80, and 100 per cent relative humidity. The results of these tests are given in Table V.

When working in these relatively short-time exposure ranges the results are not quite so clear cut as in the longer periods of time. It is apparent, however, that even 24 hours will show the difference between 0 per cent and 100 per cent relative humidities. For humidity variations from 50 to 80 per cent, the time must be lengthened somewhat, but it begins to show in from 72 to 144 hours, although even the 144-hour period is not sufficient to show the reversal of slope in the Hexa stock.

Effect of Humidity on Samples Exposed After Curing:

All of the data given in this paper so far refer to specimens which were exposed to various conditions before curing. In an earlier unpublished report, we had considered the effect of relative humidity on samples which were exposed after curing. As much of that report as is relevant to this subject will be included in this paper.

At that time we used only two stocks and made no attempt to correlate their behavior after curing with their history before curing. It is complete enough in itself to furnish us with conclusive data on the behavior of cured test slabs of these two compounds, subjected to various humidity conditions and for that reason is incorporated in this paper. These two stocks were:

	COMPOUND E	COMPOUND F
Pale Crepe	920	920
Sulfur	37	37
Hexa	28	9
XX Red Zinc Oxide	1035	224
Carbon Black	...	251
Stearic Acid	9	9

Compound E, Cured 1 hour at 40 lb., Compound F, Cured 2 hours at 40 lb.

Cured samples of these stocks were exposed for 10 and 20-day periods at 0, 50, and 100 per cent relative humidities. Table VI gives the changes in moisture content and Table VII, the effect upon tensile properties.

The tensile strength and stiffness decrease with increase in relative humidity. The samples of compound E show an increase for 20 days over the 10-day period, but this is probably a result of aging rather than humidity. The samples of compound F show a decrease.

TABLE VI.—CHANGE IN MOISTURE CONTENT CAUSED BY EXPOSING CURED SPECIMENS TO DIFFERENT RELATIVE HUMIDITIES.

Values are grams change in weight of 100-g. samples.

	RELATIVE HUMIDITY, PER CENT	10 DAYS	20 DAYS
Compound E..	0	−0.19	−0.24
	50	−0.09	−0.07
	100	.....	+1.3
Compound F..	0	−0.07	−0.02
	50	+0.14	+0.19
	100	+0.17	+0.87

TABLE VII.—EFFECT ON TENSILE PROPERTIES CAUSED BY EXPOSING CURED SAMPLES TO VARIOUS RELATIVE HUMIDITIES FOR DIFFERENT LENGTHS OF TIME.

	Exposure, Relative Humidity, per cent	Tensile Strength lb. per sq. in.		Elongation, per cent		Stress at 300 Per Cent Elongation, lb. per sq. in.		Stress at 450 Per Cent Elongation, lb. per sq. in.		Slope, lb. per sq. in.	
		10 Days[a]	20 Days[a]	10 Days	20 Days	10 Days	20 Days	10 Days	20 Days	10 Days	20 Days
Compound E...	0	3625	3750	615	615	835	935	1775	2010	940	1075
	50	3510	3725	615	630	750	895	1645	1855	845	960
	100	3500	3590	645	620	645	675	1355	1630	710	955
Compound F...	0	4405	4245	615	615	860	1025	2025	2325	1160	1300
	50	4205	3880	615	625	875	1045	2010	2355	1135	1310
	100	3995	3710	615	615	850	990	1880	2216	1030	1270

[a] Exposure period.

CONCLUSIONS AND RECOMMENDATIONS

All of the facts so far learned point very definitely to the conclusion that varying amounts of moisture and lengthening periods of time cause corresponding changes in tensile strength and stiffness in any stock.

It is true that our usual laboratory runs last only three days and that, in general, over long periods of time (such as a year), the average humidity will vary only about 40 per cent, but it is also true that a few of our recures stand from 10 to 15 days and our master batches stand from 20 to 40 days. Furthermore, relative humidity may vary almost from 0 per cent to 100 per cent in a 24-hour period and remain at its new level for 3 or 4 days; quite long enough to make a very decided change in the stock exposed during that time.

From the data obtained in this series of tests, we have concluded:

1. Humidity control is desirable.

2. A constant humidity storage cabinet is sufficient for vulcanized samples. Time of exposure is the major factor and consequently a constant humidity room for testing is not necessary because of the short time that the test specimens are exposed.

3. Since moisture changes have a very large influence on unvulcanized stocks before and after milling, a humidity controlled cabinet should be used for the storage of these stocks.

4. No control of humidity conditions is necessary during milling. Moisture is usually given off during this process at the temperature (70° C.) which is ordinarily used in our laboratory.

5. We recommend a low relative humidity as the best working condition and have chosen 0 per cent because:

(*a*) It is the easiest to maintain;

(*b*) Under this condition the total moisture content is low and therefore changes in moisture content are reduced to a minimum;

(*c*) When higher humidities are used changes in moisture content of some stocks are much more rapid than in others, and consequently, comparative results which presuppose all conditions to be equal, are an impossibility;

(*d*) Uncured stocks, especially reclaim, kept at a high[1] humidity for too long a time, tend to develop porosity upon vulcanization.

6. Even though temperature and humidity controls are adopted for the testing laboratory, a schedule for routing the samples through the laboratory in a definite time period is desirable to prevent non-uniformity through physical and chemical aging effect.

As a first step toward the control of humidity, we are installing two dry storage cabinets, one for crude rubber and master batches and the other for cured test slabs. Later, after we have proved the value of this installation, we intend to build a larger dry cabinet to store the stocks between milling and vulcanization.

Preliminary work has shown that moisture has an effect upon the abrasive qualities of tire treads. In the future this work will be amplified and the effect of moisture upon the heating up of solid tires in service will be investigated.

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[1] Van Iterson, *India Rubber Journal*, 1918, Vol. LVI, p. 423.

We have recommended a dry condition for control testing. There will be research programs in which it will probably be desirable to approximate the humidity conditions of service. In such a case the vulcanized samples could be stored at different humidities, a low humidity, for example, to approximate Arizona weather, and a high humidity to approximate the moisture conditions in damp sections of the country.

DISCUSSION

Mr. Busby. MR. H. S. BUSBY.[1]—One of the difficulties we have been experiencing in all sorts of fabrics, some of which are rubberized and otherwise coated, is to get moisture on a basis that makes it possible to say that the moisture given off either from a column of air or from any other source, like a spray, is directly applied to the fabric. In other words, we can run the fabric through the region that is supposed to be bone dry atmosphere or we can run it through a moist atmosphere, but anyone who has worked with devices for producing a humid atmosphere knows that they are not always uniform in the mill field of operation. This consideration has a very real influence in textiles as well as in rubber and in fabrics that are on the border line between the two.

Mr. Depew. MR. H. A. DEPEW.[2]—I am not quite certain as to Mr. Busby's question. Is the question "How are we certain that we had zero per cent?"

Mr. Busby. MR. BUSBY.—Yes.

Mr. Depew. MR. DEPEW.—We aspirated definite volumes of the dry air through a P_2O_5 tube and weighed it before and after. We found that a wet and dry bulb thermometer was accurate in the range between 15 per cent and 90 per cent relative humidity. We kept one in the line to make sure there was no large error, but for the final check we used the P_2O_5 tube. Where we claimed zero per cent relative humidity we were at all times within 3 per cent. For laboratory control we should be entirely satisfied with anywhere between 0 and 10 per cent because, as we have shown in the paper, in that range the amount of moisture that will be taken up is relatively small.

Mr. Merrill. MR. A. B. MERRILL.[3]—I should like to ask Mr. Depew whether he does not feel that oxidation gives him an increase in weight in his results. It is a well-known fact in aging rubber that oxidation takes place and, therefore, an increase in weight. I believe that this might influence his results somewhat.

Mr. Depew. MR. DEPEW.—The increase in weight of 100 g. of this type of rubber stock in four years of normal aging is about 4 g. That means that the increase in weight in ten days can be calculated as $\frac{1}{146}$ of 4 g. or less than 0.03 g. It certainly would not age faster at first than later on; if anything, it will go the other way. There could not be over a couple of places in the second decimal point that would be influenced by the change in weight due to aging. Furthermore, moisture was lost in the dry atmosphere, the weight was actually going down. **Mr. Depew.**

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[1] Director, A. French Textile School, Georgia School of Technology, Atlanta, Ga.

[2] Research Division, New Jersey Zinc Co., Palmerton, Pa.

[3] B. F. Goodrich Co., Akron, Ohio.

MR. STANLEY KRALL.[1]—I should like to mention that the Rubber Division of the American Chemical Society has been doing work at the U. S. Bureau of Standards along these lines on the effect of humidity on rubber testing. A preliminary report was presented at the Philadelphia meeting and Mr. J. E. Partenheimer, the chairman of the Sub-Committee on Standard Procedure for Testing Rubber Products, of Committee D-11 on Rubber Products, is keeping in touch with that work; in fact, he is the chairman of the committee that is doing this work. That work will be finished this fall and I know a very good report will be submitted. Preliminary results were very interesting, and it will be a great help in testing. **Mr. Krall.**

MR. DEPEW.—I should have mentioned that at the time of presenting the paper, I intended to. It is not referred to in the paper because we had prepared our paper before the American Chemical Society report was presented. **Mr. Depew.**

MR. H. L. CURTIS.[2]—Some two or three years ago we started to make an investigation on electrical properties of rubber compounds, and we found that we could not get results to our complete satisfaction until we had carefully controlled the moisture conditions under which the samples were kept and measured. We have improved our methods of test over these years and now our electrical tests on rubber samples are practically all made under conditions of zero humidity, after they have reached practically equilibrium condition. **Mr. Curtis.**

MR. DEPEW.—I might say that I think the most important thing in this paper is that very thing; to definitely choose conditions of humidity that will be adopted as standard. The question of whether we shall choose 0 per cent, 10 per cent, 50 per cent or 80 per cent is secondary. It is interesting to find that an electrical testing laboratory favors the same humidity (zero per cent) that we have recommended. **Mr. Depew.**

MR. KRALL.—I should like to ask Mr. Curtis how many days it takes his tests to reach equilibrium? **Mr. Krall.**

MR. CURTIS.—Five to twenty-five days. I do not have any very definite figures. **Mr. Curtis.**

MR. BUSBY.—That can be done in much quicker time if you have the proper apparatus for it. But it is a little expensive. It requires concentration and heat and moisture. **Mr. Busby.**

MR. R. R. WILLIAMS.[3]—Our experience has indicated that this element of concentration is extremely important. I should suggest **Mr. Williams.**

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[1] Fisk Rubber Co., Chicopee Falls, Mass.

[2] Senior Physicist, U. S. Bureau of Standards, Washington, D. C.

[3] Chemical Director, Bell Telephone Laboratories, Inc., New York City.

Mr. Williams. to those considering this matter that there would be an advantage in choosing a humidity that approximates normal atmospheric humidity so that much change toward equilibrium does not have to take place.

In a mass of rubber $\frac{1}{2}$-in. thick, it can be shown that six or eight months are required to reach equilibrium and the internal part of the specimen is still a part of the structure to be tested. You might get quite variable results through having a soft, distensible exterior and a dry, less distensible interior.

With regard to the matter of regulating humidities, we have found it very convenient to use a saturated salt solution which gives a vapor pressure corresponding to what we want. We have made a selection of ten or twelve of the salts so that we can obtain such solutions with quite a range of vapor pressures. By using desiccators or other form of closed vessels, we have found we could get very close approximations to the desired humidities. We have used these solutions not only with rubber, but also with textiles which show a much more rapid response.

Mr. Curtis. MR. CURTIS.—In regard to this matter of choosing humidity, I think there is one point that is decidedly in favor of zero humidity. Mr. McPherson, who has done the main part of the work on the matter to which I have referred, has found that by milling at a very high temperature he can pretty well get rid of the moisture and bring the rubber on the rolls to a condition that is very closely approaching zero humidity.

It would be much simpler and easier to bring it to an equilibrium condition on a zero per cent humidity if properly handled than practically any other humidity that might be chosen.

Mr. Depew. MR. DEPEW.—That is exactly the point we emphasize in the paper, that in one stage of rubber manufacturing the rubber is going to be dry, and that is the time when it comes off the mill. All we need to do from then on is to maintain it in that condition.

Mr. Williams. MR. WILLIAMS.—Providing one is a rubber manufacturer and not a user. In the latter case the rubber does not reach one's hands in a dry state. Also, open steam-cured goods are not dry immediately after curing.

Mr. Depew. MR. DEPEW.—In this discussion, we have been talking entirely from the viewpoint of the testing laboratory of the manufacturer. That is not so satisfactory from the viewpoint of the American Society for Testing Materials, which is largely one of specifications requirements, but I believe the consumer will be as well off with zero per cent as with any standard humidity.

RATE OF ELONGATION IN TENSION TESTS

By J. Hammond Smith[1]

Synopsis

The rate of elongation of a material under tension test is recognized as being an important factor in the interpretation of its physical characteristics.

This paper is based on a series of tests planned to show the variation of rates, throughout the critical stages, for various sizes and shapes of specimens, and various methods of gripping. The materials tested include hot-rolled, cold-rolled and cold-drawn steel.

Diagrams and tables are used to show the ratios between movement of the head of the testing machine, slipping of grips, slipping of specimen and its rate of elongation.

This investigation involves two closely related groups of main tests, which may be classified as follows:

I. General experiments with intermittent loading, to determine the relative rate of movements of the pulling head of the testing machine, the grips, and the total elongation of the specimen.

II. Non-intermittent tests involving the use of a new autographic stress-strain instrument, with time recording mechanism attached. The results show rates of elongation, at high and low speeds, compared to total load.

Principal Testing Apparatus

Standard Equipment:

A universal testing machine of the Olsen type, 100,000-lb. capacity with hand-operated poise, was used for the principal experiments.

In the experiments under Group I, Wissler wire-wound dials, reading to 0.0002 in., were used in pairs for measuring the movement of grips and total elongation of specimens. An ordinary deflectometer reading to 0.001 in. was used for measuring the speed of the pulling head of the testing machines.

Brinell machines were used in the hardness tests.

New Equipment—Autographic Stress-Strain Instrument:

The autographic stress-strain instrument has a gage length of 8 in. and is attached directly to the test specimen by means of four

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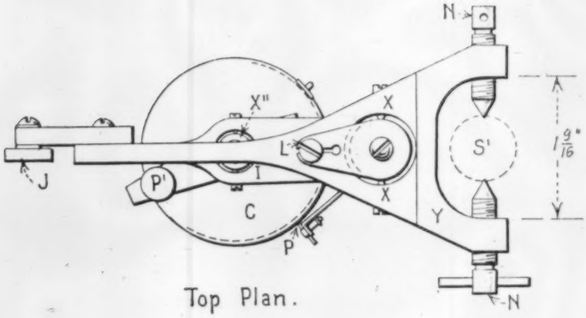
[1] Professor of Civil Engineering, University of Pittsburgh, Pittsburgh, Pa.


N
X"
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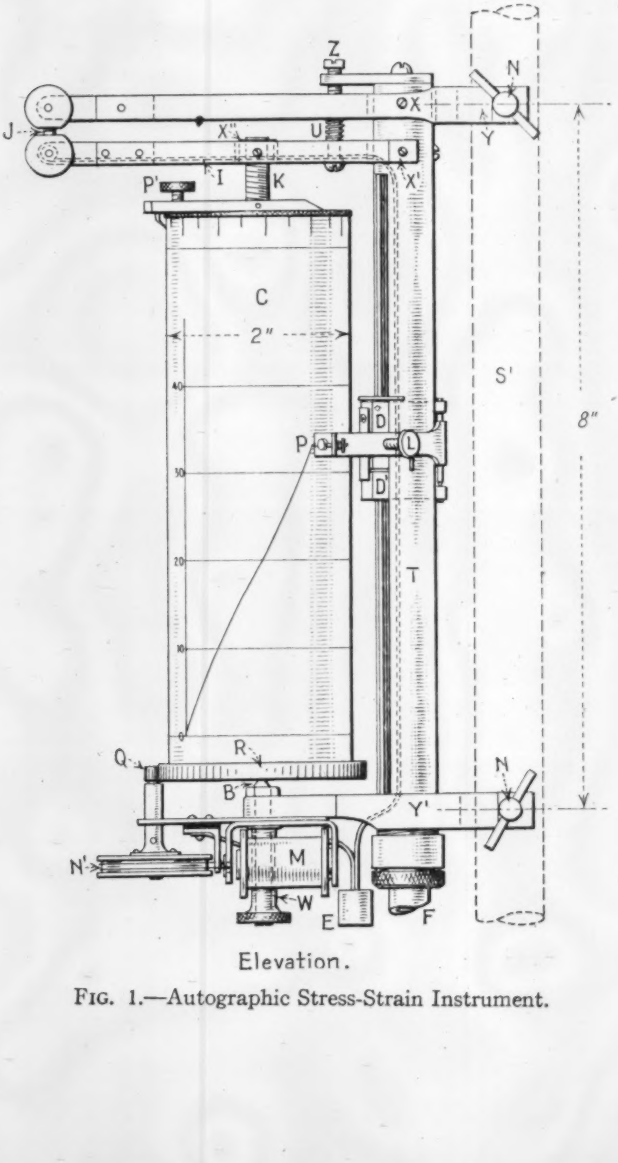
Top Plan.


Z
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0
Q
R
N
B
Y'
N'
M
W
E
F

Elevation.

FIG. 1.—Autographic Stress-Strain Instrument.

pointed contact screws. Reference to Fig. 1 will serve to explain the principal features. The frame consists of the lower yoke Y' and the slotted seamless steel tube T, rigidly connected by means of a screw thread at the lower end of the tube. The upper yoke Y is pivoted on tube T at X and carries a contact point at J. A secondary lever I is also pivoted on tube T at X'. It carries a pivoted nut X'' which engages with the left-hand cylinder screw K. It also carries an insulated contact point at J. The record cylinder C is supported on pivot B at the bottom, and at the top by means of screw K, which is attached to the top of the cylinder by means of a swivel joint and clamp P'. This swivel joint permits the adjustment of the cylinder to any convenient position, so that the autographic record may be traced at any desired location on the paper. A take-up spring U, between levers I and Y, eliminates all lost motion in the strain-measuring mechanism. An adjustable limiting screw Z serves to hold the upper yoke in proper position for attachment to the specimen. The vibratory armature of the "dead-beat" motor M, acting through the spring-balanced wrapping connectors, causes drum N' to rotate and, thus, by means of pinion Q and gear R produces a rotation of the record cylinder. The motor, with its gear Q, may be disengaged from the cylinder gear by operation of the clamp screw W. Slotted tube T serves as a guide for the pencil carriage as well as a part of the frame of the instrument. The rear arms of the carriage D, projecting through the rear slot of the tube, carry the pivots of the pencil arm, and the front projection of the carriage engages with the latch L of the pencil arm, thus permitting contact of pencil P with the record paper as desired. Vertical movement of the pencil is secured by means of a screw, centrally located within the tube T. This screw engages with a segmental nut D', attached to the pencil carriage by means of a hinged joint. Thus the carriage may be disengaged from the screw and adjusted vertically to any desired position.

By means of a special gear mechanism of simple design (attached at the rear of the main beam-supporting standard of the testing machine—not shown in Fig. 1), connecting with the screw of the pencil carriage through a specially designed flexible shaft F, the pencil is caused to move in synchronism with the poise on the weighing beam of the testing machine. Several change gears permit suitable selection of load scales. Current of 6 volts is supplied to the motor through the double plug connector E. As shown in Fig. 1, one side of the circuit is grounded on the frame of the instrument.

The form of flat grips used in the tests is illustrated in Fig. 2.

Procedure in Use of Instrument:

The specimens are inserted in the testing machine without previous marking, except as to spacing of grips. The instrument is firmly attached to the specimen by means of the four clamp screws *N*. Bringing the poise of the testing machine back to zero load brings the pencil to its zero line near the bottom of the record cylinder. A shift in rotation of the cylinder, by operation of clamp *P′* and motor drum *N′*, will establish electrical contact at *J* and thus bring the cylinder to the starting point of the record curve on the zero line. The testing machine is now started and the weighing beam carefully kept at balance. As the load increases, the recording pencil rises in synchronism with the movement of the poise, thus recording the load on the vertical scale. The elongation of the specimen is automatically recorded as the test progresses. As the specimen elongates, the contact at *J* closes and the motor instantly starts and runs for a very small fraction of a second. This causes the record cylinder to rotate slightly, carrying screw *K* with it and thereby lowering lever *I* and opening the contact points. This stops the motor instantly

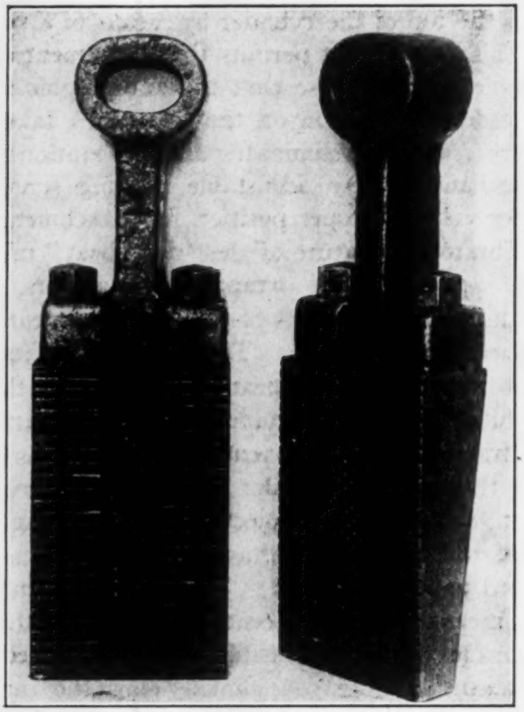

FIG. 2.—Form of Flat Grips Used in Tests.

so that there is no "coasting" of the motor. These contacts follow so rapidly and for such brief periods of time that the record line produced is a smooth curve (see Fig. 6).

Load reference lines are put on the record by bringing the poise to the desired point on the weighing beam, then rotating the cylinder with the pencil in contact. Vertical reference lines are made by running the poise out and in, or by disengaging the pencil carriage from the screw and moving it up and down as far as desired.

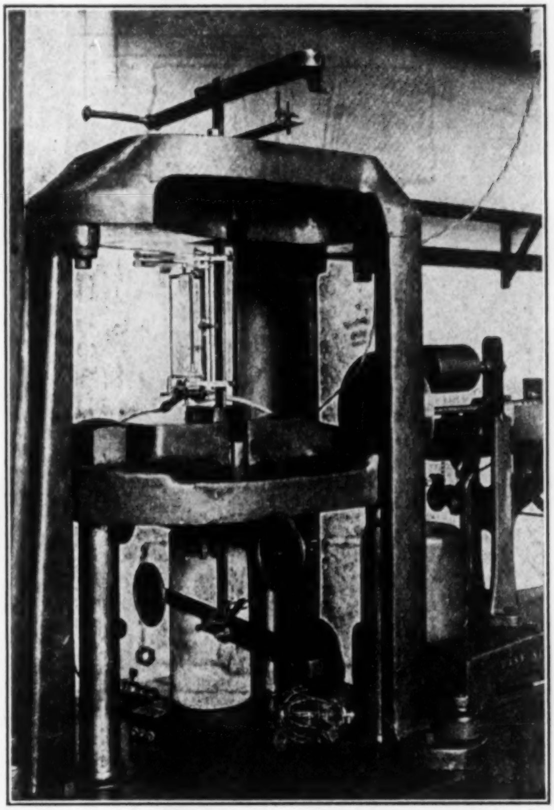

FIG. 3.—General test to determine rates of movement of head of testing machine, slipping of grips, and elongation of specimen. The autographic stress-strain instrument is shown attached to the specimen.

Tests indicate that this instrument possesses remarkable accuracy. Loads may be read from the record cards with maximum errors of about one per cent. The elongation scale is worked out so that one inch on the record represents 0.001 in. per inch of length of the specimen. The elongation error rarely exceeds 0.0001 in. in the 8-in. gage length.

Metallic-coated record paper, with a hard brass tracing point, has given excellent satisfaction on this series of tests. The improved pencil carrier contains a special "circle marker" (not shown in Fig. 1), so that any desired point may be "spotted" on the record as the test progresses. Thus, time or rate of elongation may be recorded (see Fig. 6).

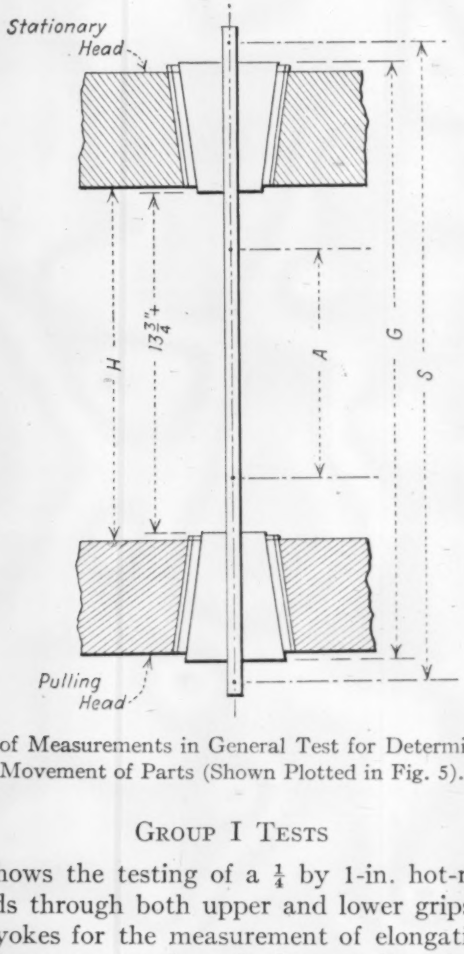

FIG. 4.—Diagram of Measurements in General Test for Determination of Rates in Movement of Parts (Shown Plotted in Fig. 5).

GROUP I TESTS

Figure 3 shows the testing of a $\frac{1}{4}$ by 1-in. hot-rolled steel bar. This bar extends through both upper and lower grips to permit the attachment of yokes for the measurement of elongation. A pair of wire-wound dials are mounted on the lower yoke, with wires extending to the upper yoke. In like manner, yokes are mounted on the upper and lower grips with dials attached to the lower yoke. Movement of the pulling head is measured by means of a deflectometer resting on the table of the testing machine and bearing on the under side of the pulling head.

The diagram shown in Fig. 4 serves to further explain the method of measuring the rates of movement in this test. Thus from variations in distance H or its equivalent, measured between the pulling head and the table of the testing machine, the rate of movement of the head may be determined. In like manner the rate of movement of the grips is determined by variations in distance G and the rate of total elongation by variations in distance S. Therefore, if H', G' and S' represent these rates respectively, the relations between them may be expressed in the equation:

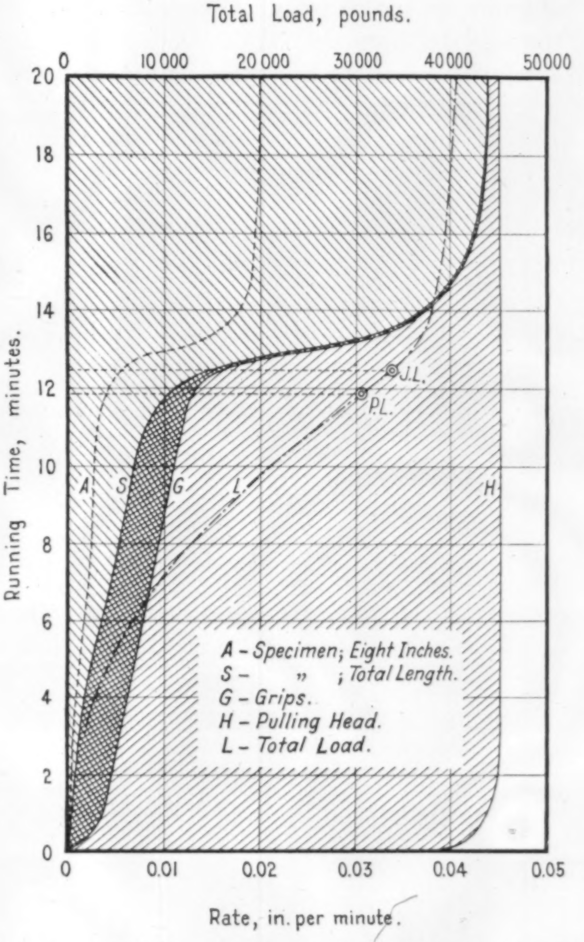

FIG. 5.—Movement of Pulling Head and Grips and Elongation of Specimen, ¾-in. Round Cold-Rolled Steel.

$$H' = S' + (G' - S') + (H' - G')$$

From this it is evident that the movement of the pulling head, H', is taken up in elongating the specimen, S', slipping of the specimen in the grips, $G' - S'$, and slipping of the grips in the pulling head, $H' - G'$.

These relations are shown graphically in Fig. 5. The horizontal distance of the curve S from the vertical zero line at the left shows the rate of total elongation of the specimen; the distance between curves S and G shows slipping of the specimen in the grips, and the distance between curves G and H shows slipping of the grips in the pulling head.

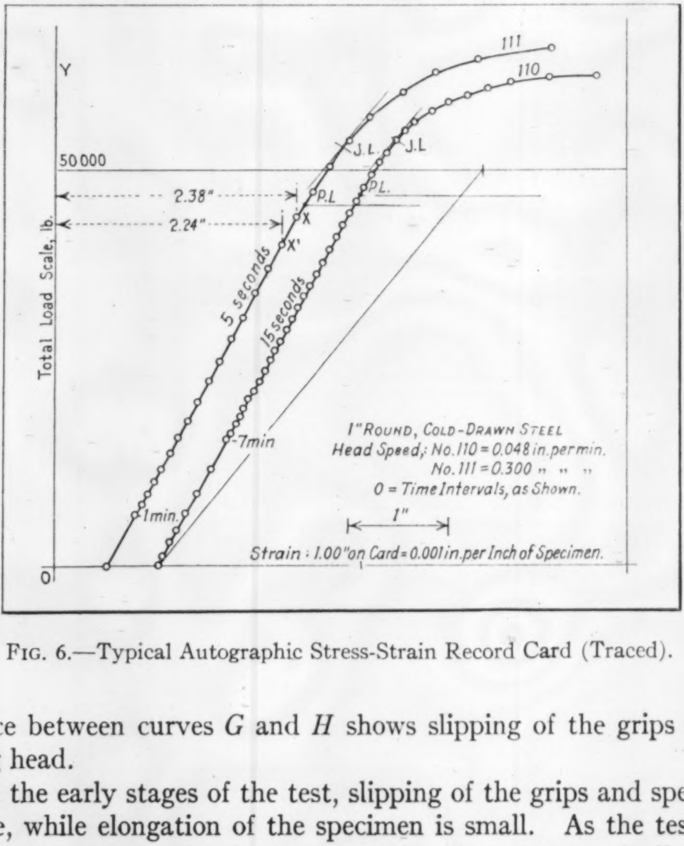

FIG. 6.—Typical Autographic Stress-Strain Record Card (Traced).

In the early stages of the test, slipping of the grips and specimen is large, while elongation of the specimen is small. As the test progresses, the rate of slipping of the grips decreases gradually until the proportional limit (P.L.) of the specimen has been reached. Then a rapid change in these rates takes place. The slipping of grips and specimen decreases to near zero and the movement of the pulling head is almost entirely taken up in the elongation of the specimen. For clarity, the areas representing the various movements and elongation of specimen are shaded.

The Johnson elastic limit of the material is indicated by the letters *J. L.* Curve *A* was plotted from the autographic stress-strain record. The total load curve is also included in this diagram, so that it may be inspected for relation between loading and all other factors involving movement and elongation.

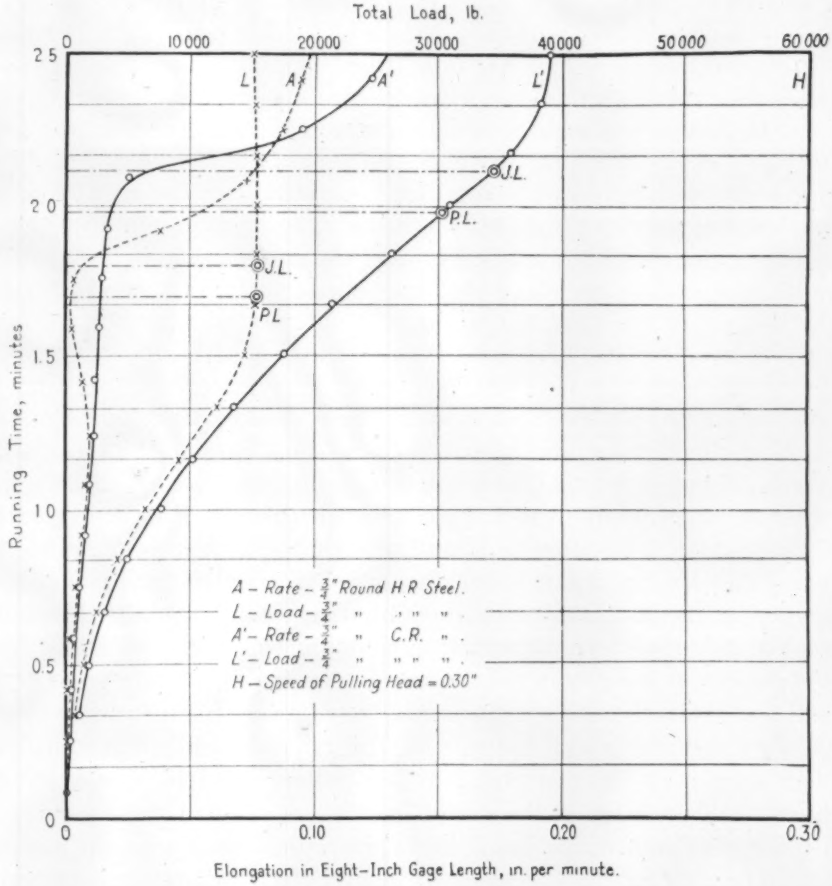

FIG. 7.—Rate of Elongation With Total Load. Comparison of Hot-Rolled and Cold-Rolled Steel Bars. Continuous Loading at High Speed.

GROUP II TESTS

The autographic stress-strain instrument, with time-recording pencil attached, was used in these experiments. The record, therefore, consisted of load, elongation and time, as shown by the autographic cards, one of which is reproduced in Fig. 6.

The rate of elongation is determined by scaling the horizontal (elongation) distance between consecutive time record points (circles). For example, distance of circle X from the vertical reference line Y is 2.38 in. and the distance of circle X' is 2.24 in., therefore, the elongation in the interval of time (5 seconds) between these two circles is $2.38 - 2.24 = 0.14$ in. One inch on the card represents 0.001 in. per inch of gage length of the specimen. Therefore, the average rate of elongation during this 5-second interval is $(0.14/1000) \times 8 \times 12 = 0.01344$ in. per minute for the 8-in. gage length. In the same manner rates for other intervals shown on the card may be determined.

In plotting (as shown in rate diagram, Fig. 7) these values are set on the median lines between the time values corresponding to the points measured. The total load is scaled from the card at each of the time points and plotted accordingly.

Figure 7 shows the relative rates of elongation, with corresponding total load curves, for $\frac{3}{4}$-in. round hot-rolled and cold-rolled steel bars, with speed of the pulling head at 0.300 in. per minute. Curves A' and L' may be compared with curves A and L of Fig. 5, both tests being made on the same size and quality of material—$\frac{3}{4}$-in. round cold-rolled steel. The latter were made at low-speed intermittent loading, the former at high-speed continuous loading. In each of these diagrams, it will be noted that for the $\frac{3}{4}$-in. cold-rolled bars the rate of elongation suddenly increases at or near the proportional limit point. This increase is still more rapid at the Johnson limit point and reaches a maximum change above that point. The cause and effect of this change may be more clearly understood by a study of Fig. 5. It will be noted that the slipping of grips uniformly decreases until the proportional limit is reached. At this point the ratio of resistance of specimen and resistance of grips to slipping changes suddenly, and the rate of the grips decreases accordingly. This results in the movement of the pulling head being taken up mainly in elongation of the specimen.

Again referring to Fig. 7, it will be noted that curve A (rate of elongation of a $\frac{3}{4}$-in. hot-rolled steel bar) decreases to near zero at the proportional limit. This is caused by the fact that these bars were straight and held in V-grips, which resulted in the bars passing the elastic limit, first at the grips and later at mid-section, thus giving a distorted value of the rate in the 8-in. gage length. This, however, shows typical results from this character of tests.

The results of a series of tests under Group II are shown in Table I. These were straight bars of uniform cross-section end to end and 20 in. long. The grips were set 12 in. apart, flat grips being used for all flat specimens and V-grips for round specimens.

TABLE I.—RATE OF ELONGATION OF STRAIGHT STEEL BARS.

A = Rate of Elongation in 8-in. gage length in inches per minute

HOT ROLLED

Group No.	Cross-Sectional Dimensions, in.	Cross-Sectional Area, sq. in.	Brinell Hardness Number	Number of Grip Liners in Each Head	Total Load at Elastic Limit, lb.	Maximum Rate of Elongation below Elastic Limit, A, in. per min.	$\frac{H}{A}$
SPEED OF PULLING HEAD (H) = 0.048 IN. PER MINUTE							
50	$\frac{1}{4}$ by 1	0.250	152	4	11 700	0.0041	11.70
60	$\frac{1}{4}$ by $1\frac{1}{2}$	0.375	143	4	18 000	0.0036	13.32
1	$\frac{3}{8}$ by $1\frac{1}{2}$	0.563	99	3	18 500	0.0024	20.00
70	$\frac{1}{2}$ by $1\frac{1}{2}$	0.750	102	4	24 000	0.0022	21.81
30	$\frac{3}{4}$-in. round	0.442	92	5	14 050	0.0018	26.67
40	1-in. round	0.785	115	4	27 900	0.0013	36.95
SPEED OF PULLING HEAD (H) = 0.300 IN. PER MINUTE							
50	$\frac{1}{4}$ by 1	0.250	152	4	11 950	0.0250	12.00
60	$\frac{1}{4}$ by $1\frac{1}{2}$	0.375	143	4	18 600	0.0250	12.00
3	$\frac{3}{8}$ by $1\frac{1}{2}$	0.563	99	3	19 650	0.0154	19.45
70	$\frac{1}{2}$ by $1\frac{1}{2}$	0.750	102	4	24 700	0.0139	21.66
30	$\frac{3}{4}$-in. round	0.442	92	5	15 000	0.0113	26.50
40	1-in. round	0.785	115	4	28 900	0.0082	36.56

COLD DRAWN

Group No.	Cross-Sectional Dimensions, in.	Cross-Sectional Area, sq. in.	Brinell Hardness Number	Number of Grip Liners in Each Head	At Proportional Limit			At Johnson Elastic Limit		
					Total Load, lb.	Rate of Elongation, A, in. per min.	$\frac{H}{A}$	Total Load, lb.	Rate of Elongation, A, in. per min.	$\frac{H}{A}$
SPEED OF PULLING HEAD (H) = 0.048 IN. PER MINUTE										
120	$\frac{1}{8}$ by $\frac{1}{2}$	0.063	187	5	4 950	0.0082	5.85	5 625	0.0101	4.75
130	$\frac{1}{8}$ by 1	0.125	156	5	7 250	0.0057	8.41	8 200	0.0078	6.15
140	$\frac{1}{4}$ by 1	0.250	137	4	12 150	0.0041	11.70	13 950	0.0067	7.17
150	$\frac{1}{4}$ by $1\frac{1}{2}$	0.375	146	4	......	......	.....	20 700	0.0052	9.23
170	$\frac{1}{2}$ by $1\frac{1}{2}$	0.750	163	4	39 500	0.0030	16.00	46 800	0.0043	11.15
100	$\frac{3}{4}$-in. round	0.442	137	5	20 650	0.0028	17.10	22 900	0.0041	11.70
22[a]	$\frac{3}{4}$-in. round	0.442	191	4	30 500	0.0036	13.32	34 300	0.0046	10.43
110	1-in. round	0.785	170	4	46 800	0.0022	21.81	53 900	0.0029	16.53
SPEED OF PULLING HEAD (H) = 0.300 IN. PER MINUTE										
130	$\frac{1}{8}$ by 1	0.125	156	5	7 350	0.0394	7.61	7 800	0.0451	6.65
140	$\frac{1}{4}$ by 1	0.250	137	4	12 350	0.0281	10.68	14 300	0.0404	7.42
150	$\frac{1}{4}$ by $1\frac{1}{2}$	0.375	146	4	......	......	.....	20 400	0.0298	10.06
170	$\frac{1}{2}$ by $1\frac{1}{2}$	0.750	163	4	39 000	0.0192	15.63	46 300	0.0281	10.67
100	$\frac{3}{4}$-in. round	0.442	137	5	21 350	0.0158	19.00	23 350	0.0249	12.05
22[a]	$\frac{3}{4}$-in. round	0.442	191	4	31 000	0.0204	14.70	34 600	0.0170	17.65
110	1-in. round	0.785	170	4	45 600	0.0154	19.48	52 800	0.0182	16.49

[a] Cold-Rolled.

The number of grip liners in each head of the testing machine is given, as this is undoubtedly a factor in the rate of elongation.

In the hot-rolled specimens, the rate of elongation reaches a comparatively definite maximum below the proportional limit, but drops to near zero at this point. Therefore, the maximum values are given in the table.

Rates for the cold-drawn and cold-rolled steels continue to increase as the stress rises through the proportional and Johnson limits (see Fig. 7). Therefore, results are given for both limits. For the proportional limit, rates were taken just below that point and for the Johnson limit, average rates were measured.

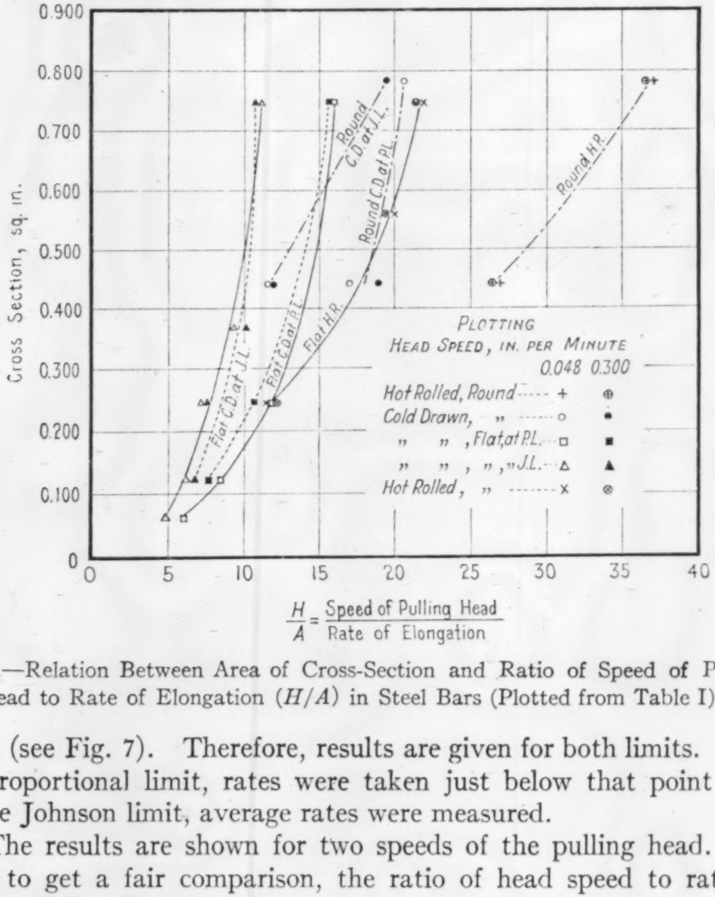

FIG. 8.—Relation Between Area of Cross-Section and Ratio of Speed of Pulling Head to Rate of Elongation (H/A) in Steel Bars (Plotted from Table I).

The results are shown for two speeds of the pulling head. In order to get a fair comparison, the ratio of head speed to rate of elongation (H/A) is shown.

CONCLUSIONS

Data from Table I were used in plotting the diagram shown in Fig. 8. It will be noted that the curves run downward to the left, toward zero, which means that if the area of cross-section is small, the rate of elongation is large, and *vice versa*. The ratio H/A is the reciprocal of the fraction of movement of the pulling head used in elongating the gage length of the specimen. For example, in Table I, Group No. 40, hot rolled, the area of section is 0.785 sq. in. and $H/A = 0.048/0.0013 = 36.95$. This shows that the head is moving 36.95 times as fast as the 8-in. gage length is elongating. Taking the other extreme from Group No. 120, cold drawn, the area of section is 0.063 sq. in., and at the proportional limit $H/A = 0.048/0.0082 = 5.85$. The head is moving only 5.85 times as fast as the specimen is elongating. The small specimen is, therefore, elongating 36.95/5.85 = 6.31 times as fast as the large one, although the speed of the pulling head is the same in each case.

By reference to Table I and Fig. 8, it will be seen that the ratio of speed of pulling head to rate of elongation of specimen (H/A) is fairly uniform for the two speeds. Therefore, in general, it appears that in any test of this character, the rate of elongation will be directly proportional to the speed of the pulling head, but will vary widely with the cross-sections of the test specimens.

The author considers these tests as being only a brief introduction to this important subject, the complexity of which demands much further careful research.

Acknowledgment.—The author acknowledges the highly valued cooperation of the Jones & Laughlin Steel Corporation in furnishing the major portion of the specimens for these tests.

DISCUSSION

Mr. Moore. MR. H. F. MOORE.[1]—Mr. Smith's paper has brought out the fact very sharply that the motion of the heads of the testing machine is many times (twenty times in one instance) the elongation of the specimen. This raises the question whether the custom of specifying speeds of head of testing machine is a satisfactory one, or whether an attempt should be made to specify rate of actual stretch of specimen or rate at which load comes on the specimen. The speed of head of machine is specified because for the ordinary screw-power machine it is conveniently measured. Rate of actual stretch of specimen or of application of load is less easily measured, but Mr. Smith's apparatus is suggestive of possibilities of making such a measurement feasible.

Mr. Lynch. MR. T. D. LYNCH.[2]—Fifteen or twenty years ago I had occasion to make tests in a study of the effects of speed of testing different kinds of steel. The test specimens used were of the A.S.T.M. standard ½-in. diameter 2-in. gage length specimen with ends threaded. The speed of head of testing machine running without load was first determined. The actual elongation between the 2-in. gage points of the test specimen was observed with the same speed of motor as when running idle. The speed ratios of head of machine and elongation of gage length of the test specimen were 10 to 1.

Mr. Templin. MR. R. L. TEMPLIN.[3]—Not long ago I had the pleasure of seeing Mr. Smith's extensometer in operation and as a result of the demonstration was quite impressed with the ingenuity of its well-worked-out details, as well as with its usefulness in the testing of materials.

The more general use of such an instrument would certainly give us more definite information concerning the effects of speed of testing on the tensile properties of materials which with the usual methods of testing may be seriously affected by movement of the specimens in the grips or of grips in the testing machine heads. The results obtained by Mr. Smith would seem to emphasize the necessity for giving more attention to grip design so that the speed of the testing machine head would bear a closer relation to rate of elongation of specimen. In certain kinds and tempers of non-ferrous metals the speed of testing has a very appreciable influence on the mechanical

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[1] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] Manager, Materials and Process Engineering Department, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[3] Chief Engineer of Tests, Aluminum Co. of America, New Kensington, Pa.

Mr. Templin. properties, so that concordant results from different laboratories on testing machines can be obtained only when the rate of elongation is nearly constant.

In criticism of the instrument described, it would seem that its usefulness would be much enhanced if it could be used on shorter or various gage lengths, say 2 in. to 8 in., and if it were light enough to be readily applied to and used on small diameter or thin specimens. To accomplish this the writer would suggest making use of the recording drum on an autographic testing machine by using two "dead-beat" motors, one to actuate the drum and the other only the electrical contact mechanism, which would of course be on the test specimen. That is, the curve-drawing mechanism, the load lead screw and recording drum would be on the testing machine, leaving only the deformation-measuring or controlling mechanism on the specimen. The two "dead-beat" motors would be connected in parallel in such an arrangement.

This idea involves some rather important details but enough work has been done on it to indicate that it can be worked out satisfactorily.

Mr. Smith. MR. J. HAMMOND SMITH[1] (*author's closure by letter*).—The idea of separating the mechanism of the instrument into two elements—elongation or contact mechanism on the specimen and recording drum on the frame of the testing machine—was studied by the author but abandoned, for the time being, on account of the apparent impossibility of keeping two motors of this "dead-beat" type in synchronism.

An instrument of the general type illustrated in this paper has been designed for tests in compression as well as tension and adjustable for various gage lengths ranging from 2 in. to 8 in. This instrument has not been built but the author is reasonably certain that it is entirely practical.

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[1] Professor of Civil Engineering, University of Pittsburgh, Pittsburgh, Pa.

RECENT DEVELOPMENTS AND APPLICATIONS OF THE ELECTRIC TELEMETER[1]

By O. S. Peters[2]

Synopsis

In 1923, an electric telemeter, developed at the U. S. Bureau of Standards, was described before the annual meeting of the Society.[3] Since that time progress has been made in the further development and in practical applications of this instrument which the paper describes.

Photographic records are included from apparatus devised to produce mechanical displacements of widely different frequencies which are shown on the same film with their reproduction by the telemeter. These serve to indicate the accuracy of the reproduction, and also to point out errors which may arise because of inertia effects in measuring strains due to rapidly changing loads. There are also shown a number of telemeter records from different applications, such as impact measurements on street car rails and acceleration stresses in coal mine hoisting cable.

The paper further describes the telemeter cartridge used for measuring strains in mass concrete at the Stevenson Creek Arch Dam and at other places. A description of a telemeter pressure gage for fluid pressures ranging up to 40,000 lb. per sq. in. is also given.

In 1923, an electric telemeter developed at the U. S. Bureau of Standards was described before the Society.[3] Since that time progress has been made in the further development and in practical applications of this instrument which it is the object of this paper to describe.

The primary purpose of the telemeter is to measure strains or forces arising in a structure from either slow or rapid changes of load and indicate or record them at a distance with sufficient accuracy for engineering purposes. This remote reading and recording feature is very useful in many cases, a usefulness which is greatly augmented by the fact that simultaneous readings or records may be made of strains at a number of widely separated points.

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[1] Published by permission of the Director of the U. S. Bureau of Standards, Department of Commerce, Washington, D. C.

[2] Electrical Engineer, U. S. Bureau of Standards.

[3] O. S. Peters and R. S. Johnston, "New Developments in Electric Telemeters," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 592 (1923).

The essential part of the apparatus consists of one or more small carbon resistors mounted within a metal frame arranged so as to be deformed elastically by the strain or force to be measured in such a way as to change the pressure upon the carbon and thus change its electrical resistance. This change of resistance is utilized in a wheatstone bridge circuit to indicate or record the effect quantitatively.

In the practical embodiment of the apparatus, the carbon resistors form the variable arm, or arms, of the wheatstone bridge, the fixed arms of which are mounted in a panel of suitable design. Connection between the carbon resistors and panel is made by a 3-conductor copper cable which may be of any length required. In place of the suspension type of galvanometer ordinarily used in the wheatstone bridge a milliammeter is substituted for visual readings and a high sensitivity oscillograph element with a mirror and beam of light for recording on film or bromide paper. By combining a number of wheatstone bridge circuits electrically in parallel, multiple units are built up of as many as twelve elements.

The carbon resistor instruments so far developed are included in two types: namely, (1) the double resistor type, and (2) the single resistor type. The double resistor type has the advantage of a calibration curve which is substantially a straight line, and is compensated in a large degree for temperature effects. The single resistor type exhibits a calibration curve which resembles a portion of a parabola and is affected in considerable measure by temperature, but for a number of uses these features do not constitute a serious disadvantage.

THE DOUBLE RESISTOR TYPE

New Developments:

The essential features of the double resistor type of telemeter have not been changed since the instrument was described previously,[1] but improvements have been made to increase the stability under shock, and the accuracy, especially in the measurement of strains due to impact.

The chief improvement has been made in the carbon resistor, which has been changed from a 1-ohm stack of annular rings of rather soft carbon under about 5 lb. initial pressure, to a 50-ohm stack of very hard carbon of the same dimensions under about 20 lb. initial pressure. The cross-sectional area is approximately 0.113 sq. in. so the unit pressure is nearly 180 lb. per sq. in.

This increase of pressure has increased the stability to a point where a force of 1000 g. (2.2 lb.) can be applied transversely at the mid-point of one stack without altering the indications of the instrument by more than 3 per cent. The weight of the carbon stack with its end plates is approximately 3 g. Hence, a transverse force of 1000 g. produces an effect which is roughly equivalent to that produced by a transverse acceleration due to shock of 330 times gravity. In the case of shock, however, either transverse or longitudinal, both stacks are affected somewhere near equally, and since the effects in the wheatstone bridge circuit are opposed to each other, they tend to neutralize. The error introduced by moving the gage rapidly about, as in measuring vibratory strains, is therefore small unless the frequency of the vibration and its amplitude both become much greater than is likely to be met with in engineering structures.

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[1] O. S. Peters and R. S. Johnston, "New Developments in Electric Telemeters," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 592 (1923).

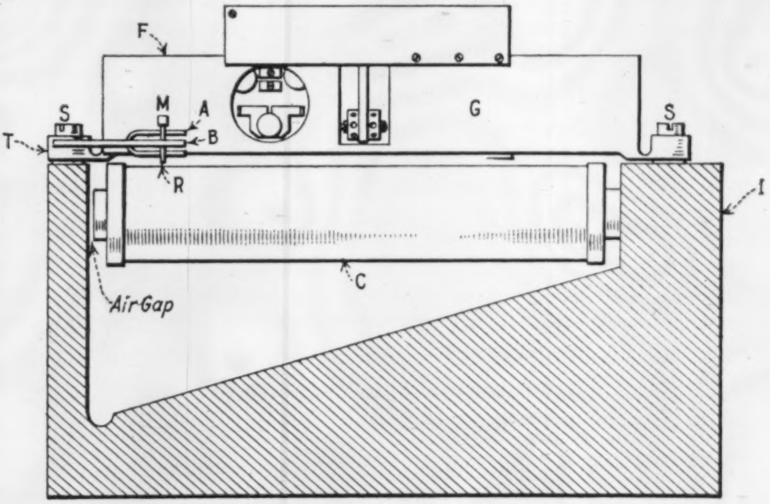

FIG. 1.—Diagram of Double Resistor Type of Electric Telemeter.

Marked improvements have also been made in the frames for mounting the carbon resistors, in the direction of lightness, rigidity, and shorter gage lengths. The gage length which appears to have the greatest usefulness is 2 in. The frame can be adapted, however, to gage lengths from $\frac{1}{2}$ in. upward. Other improvements have been made in the panel and recorder, principally in the oscillograph elements as regards damping and stability of the sensitivity during transportation.

Reproduction of Strain Wave-Shape:

When a train passes over a steel bridge a vibratory strain is produced which may have harmonics in it ranging up to several hundred cycles per second, depending upon the dimensions of the particular member considered and its loading. If a carbon resistor gage is clamped to the member the strain will be communicated to the carbon resistors. The resistance of these will be changed and destroy the balance of the corresponding wheatstone bridge circuit, causing the oscillograph mirror to deflect. This deflection is traced upon a moving photographic film which is later developed and scaled to determine the amount of the strain at any particular time during the progress of the record.

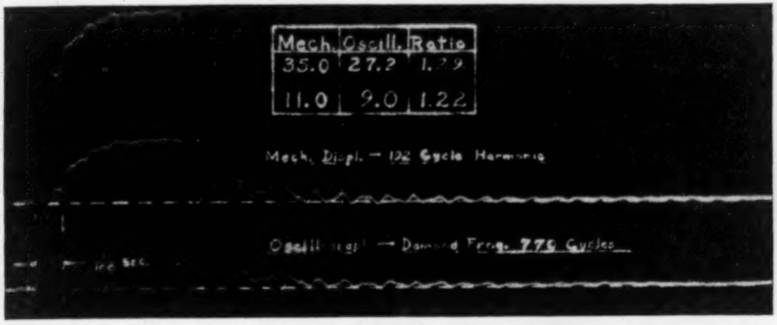

FIG. 2.—Mechanical Displacement Recorded by Double-Resistor Telemeter.

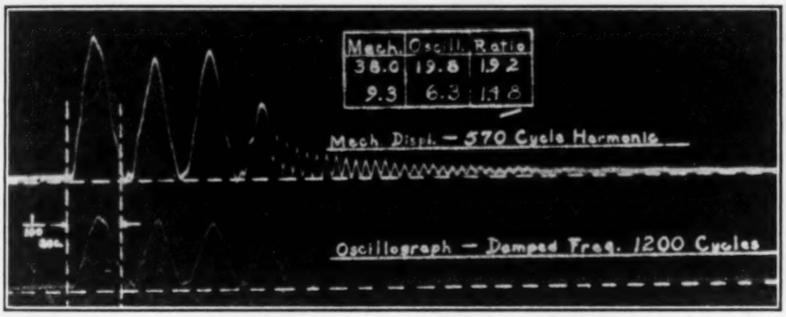

FIG. 3.—Series of Mechanical Displacements Recorded by Double-Resistor Telemeter.

In this connection the question naturally arises as to whether a trace of this kind, produced as a result of two distinct mechanical motions of apparatus placed some distance apart and connected only by wires with electric currents flowing in them, will be a true representation of the change of strain in the member, particularly if the change is rapid.

In order to determine this point a piece of apparatus shown diagrammatically in Fig. 1 was devised. In this figure, *I* represents a U-shaped piece of steel, the upper ends of which can be deflected by means of the magnetizing coil *C* which, when energized, exerts a pull across the air gap. By applying alternating current of different frequencies to the coil, and by changing the stiffness of the steel piece *I*, a variety of mechanical vibrations can be set up, of either regular or irregular character.

The carbon resistor gage *G* is clamped by screws *S* to the opposite arms of the vibrating member, so the deflections are applied to the thrust rod *T*, and move it relatively to the main frame *F*. This relative motion between *T* and *F* is recorded electrically on film by means of the carbon resistors mounted within the gage frame, and the oscillograph element. At the same time the actual mechanical motion of *T* relative to *F* is recorded on the same film by the mirror *M* which is actuated by roller *R* and parts *A* and *B*, one of which is attached to *T* and the other to *F*.

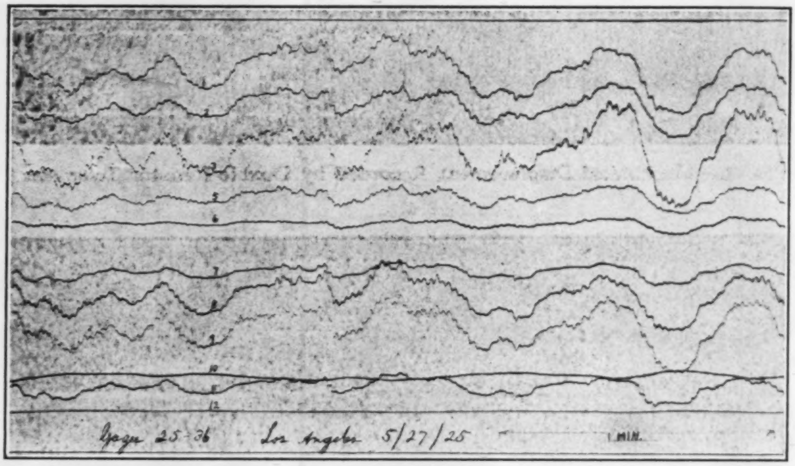

FIG. 4.—Double-Resistor Telemeter Record from a Rigid Airship in Flight.

In Figs. 2 and 3 are shown two photographic records taken with this apparatus. That in Fig. 2 is of a mechanical displacement which took place in a period of 0.05 second with a superimposed vibration having a frequency of about 192 cycles per second. That in Fig. 3 is of a succession of mechanical displacements which took place in about 0.01 second each with a superimposed vibration having a frequency of about 570 cycles per second. If these records are followed point by point it will be observed that each ripple on the mechanical wave is reproduced by the carbon resistors and the oscillograph.

Whether the reproduction is a true one, of course, depends upon whether the deflection of the oscillograph is at all points proportional to the mechanical displacement. A convenient means for determining

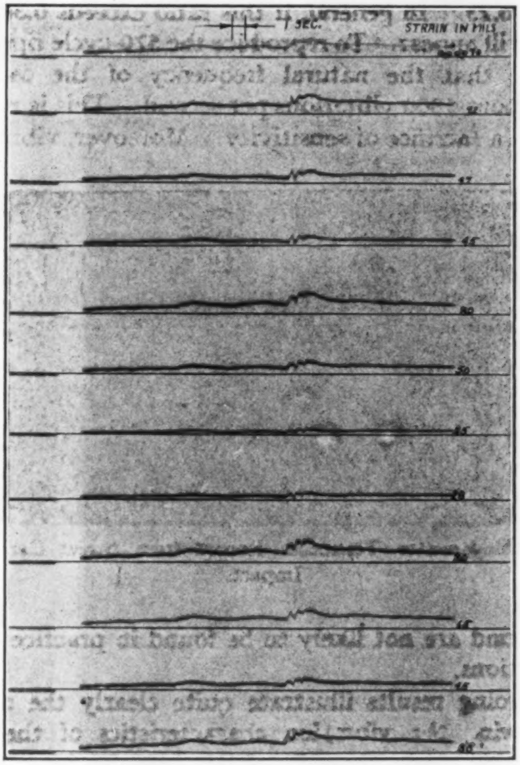

FIG. 5.—Double-Resistor Telemeter Record from a Highway Bridge Slab of Concrete.

this is to measure the relative heights of some of the corresponding points on the two traces in each record. If the ratio of these for a low frequency and a much higher one are equal, or nearly so, it is safe to assume that the reproduction is a true one. In the case of Fig. 2 the ratio of the large deflections is found to be 1.29, while for the 192-cycle ripple it is 1.22. These values check within the possible errors of measurement and indicate that the reproduction is sufficiently exact for the uses likely to be made of it.

In the case of Fig. 3, however, the ratio of the large deflections is 1.92, while that of the 570-cycle ripple is 1.48, which indicates that the oscillograph deflection for the high-frequency ripple is exaggerated by about 23 per cent. This exaggeration comes from the fact that the frequency of the measured vibration is too near that of the oscillograph element, which is 1200. The ratio of frequencies is about 0.48, whereas, in Fig. 2 it is 0.25. In general, if this ratio exceeds 0.30, appreciable exaggeration will appear. To reproduce the 570-cycle ripple accurately would require that the natural frequency of the oscillograph be increased to about 1800 vibrations per second. This is readily accomplished, but at a sacrifice of sensitivity. Moreover, vibrations as high as 570 per second are not likely to be found in practice except under unusual conditions.

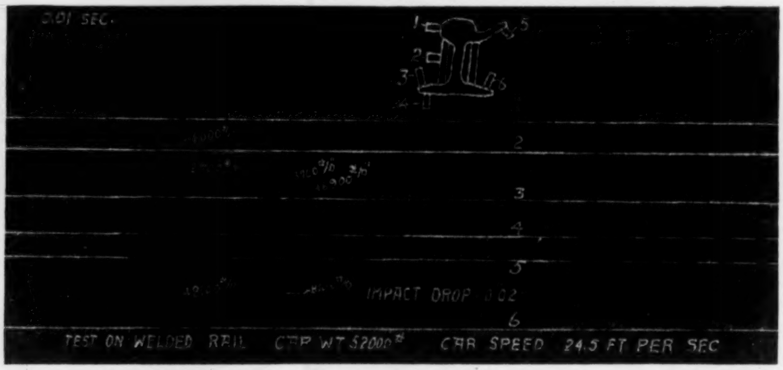

FIG. 6.—Double-Resistor Telemeter Record from Street Car Rail Under Impact.

The foregoing results illustrate quite clearly the necessity, not only for knowing the vibration characteristics of the instruments used in measuring strains from live loads, but also for having a record from which the frequency of the vibrations can be determined. Without these there can be no certainty that results represent actual effects.

Typical Records Obtained with Double Resistor Telemeter:

In Figs. 4 to 8 inclusive are shown a number of telemeter records, not with the object of analyzing the structural stresses here, but to indicate the wide variety of conditions met with in measuring strains due to live loads. On each record the time scale is indicated, and on some of them the actual stresses.

Figure 4 is from a rigid air-ship in flight, with 12 carbon resistor gages attached to the keel members and girders. The large waves of stress come from pitching of the ship fore and aft in "bumpy" air. The smaller ripples come mostly from motor vibrations. The maximum change of stress indicated is about 4000 lb. per sq. in. in duralumin.

Figure 5 is from a highway bridge slab of concrete supported by steel I-beams. In this case a loaded motor truck was driven over an obstruction on the bridge. The maximum change of stress shown for the I-beams is only about 3000 lb. per sq. in. It should be noted, however, that this was a test bridge made much stronger than the general run of existing bridges.

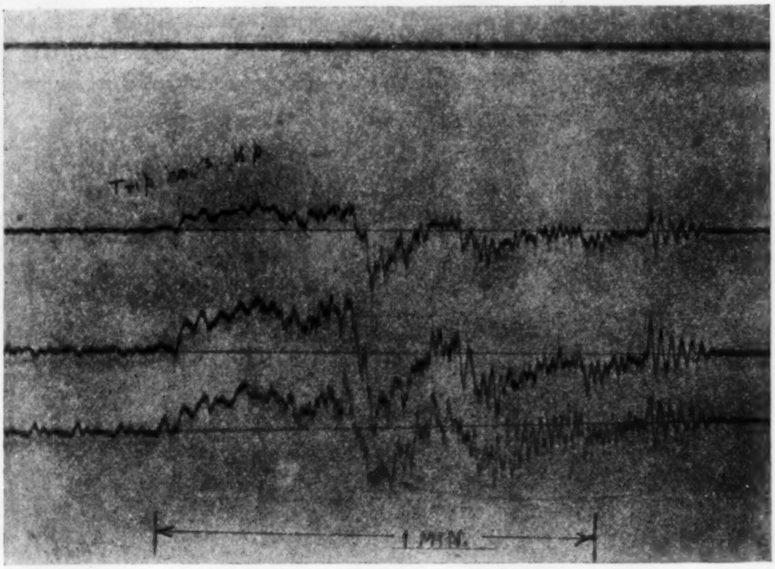

FIG. 7.—Double-Resistor Telemeter Record from $1\frac{1}{2}$-in. Rope of a Coal Mine Hoist.

Figure 6 is from a street car rail under impact from the wheels of a car. Here the wheels were passed over a shim 24 in. long which had a gradual slope at the receiving end, and an abrupt drop of 0.02 in. at the run-off end.

Figure 7 is from the $1\frac{1}{2}$-in. rope of a coal mine hoist just above the cage. The three traces are from the same length of rope, with the gages placed 120 deg. apart about the circumference. The total dead load on the rope at the time this record was taken was 21,600 lb. The total travel of the cage was about 500 ft. The record indicates an increase of stress on the rope for this particular occasion of about 30 per cent due to initial upward acceleration, and also the fact that periodically the cage travelled faster than the circumference of the winding drum.

Figure 8 shows pressure and deflection in a test of a mine stopping slab. The pressure was produced by burning black powder in a chamber behind the slab. The differences in pressure shown were due mainly to friction losses in the pipes leading from the chamber to the telemeter gages and to leakage of gas rather than actual pressure differences within the chamber. The chief point of interest is the manner in which the slab withstood the maximum pressure for nearly one second before it blew out.

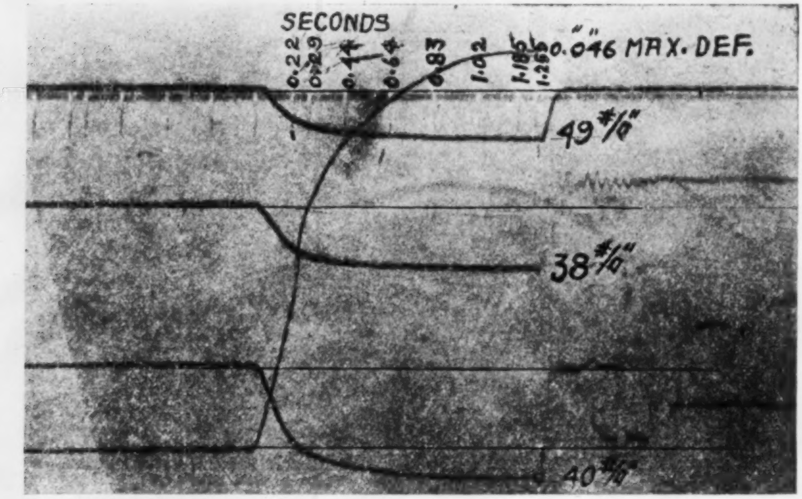

FIG. 8.—Double-Resistor Telemeter Record from Test of a Mine-Stopping Slab.

SINGLE RESISTOR TYPE

New Developments:

In connection with the tests of the Stevenson Creek Experimental Arch Dam in California, a measuring instrument was needed which could be embedded in the concrete at any point, and which would give indications at the surface of the strains under changes of load, moisture and temperature. This need led to the development of a cartridge telemeter. In its present form it can be embedded in the concrete or attached to the surface under water. Difficulty was experienced at first in sealing these cartridges against water, but this has been overcome to such an extent that it is felt that the latest type will give reliable indications for a period of years.

A further development is a pressure gage for fluid pressures which may range up to 40,000 lb. per sq. in.

Cartridge Telemeter:

The essential features of the cartridge telemeter are shown in Fig. 9. Here *A* is a steel frame cut from a piece of drill rod. The carbon resistor *E* is mounted within a rectangular portion of the frame the ends of which are slightly flexible. Parts *B* and *C* are flanged bushings threaded into the frame *A*. Part *D* is a steel cover tube. At *F* is a resistance thermometer which consists of a coil of copper wire on a spool surrounding the carbon resistor. It is proportioned to give a change of 2 ohms per 1° C. At *S* the tube is sealed to the frame with a flexible joint made with asphalt and rubber tubing. At *L* are three wires enclosed in a rubber jacket, one of which is common to the stack and coil. The point of entrance of these wires is also sealed to prevent entrance of moisture. Under test these seals have withstood 750 lb. per sq. in. hydrostatic pressure for two hours without leakage. A cartridge similarly sealed, but without the rubber tube shown at *S*, has been kept in saturated concrete for $2\frac{1}{2}$ years without showing effects from moisture. From these tests it appears that the principles involved are correct, but success in sealing a large number of cartridges depends upon the care used in sealing each one.

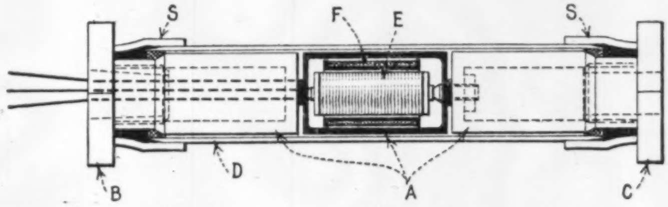

FIG. 9.—Diagram of Single Resistor Type of Cartridge Electric Telemeter.

The tube *D* is painted and wrapped with tape to prevent bond with the concrete and a consequent reinforcing action. Flanges *B* and *C*, however, are bonded to the concrete and move with it. Consequently, any relative motion between *B* and *C* is communicated to the carbon resistor, which causes its resistance to change. From this change of resistance the corresponding change of length is determined by reference to a calibration curve which must be obtained for each cartridge before it is embedded.

In Fig. 10 is shown a typical calibration curve. It will be noted from this that the range of each cartridge is about 0.006 in. The total change of resistance is from 22 to 75 ohms, so there is no need for highly accurate measurements of resistance to make sufficiently accurate determinations of change of length. This permits rapid readings. A portable wheatstone bridge is quite suitable for these readings, such as a Leeds and Northrup Type "S" Testing set.

In adjusting the cartridge prior to embedding it, allowance must be made for shrinkage of the concrete in setting, otherwise it may be that when setting is complete the resistance of the cartridge will be found outside the range of the calibration. As a rule the adjustment is made near to the top of the range, that is, a cartridge having a maximum resistance of 75 ohms is adjusted to about 65 ohms. The decrease due to shrinkage then brings the adjustment near the midpoint of the range.

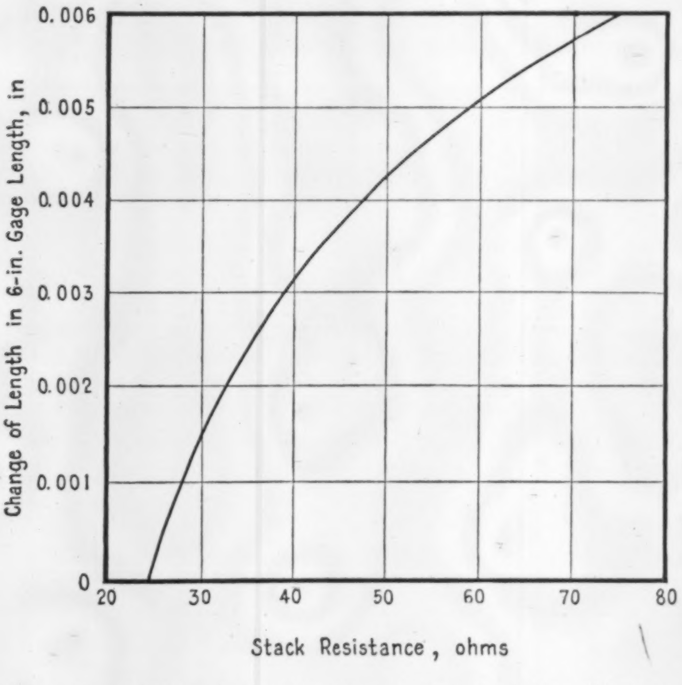

FIG. 10.—Typical Calibration Curve of a Cartridge Electric Telemeter.

A temperature correction is required because of the unequal thermal expansion of carbon, steel and concrete. This correction for a 6-in. cartridge is 0.00008 in. per 1° C.

The errors of measurement are not likely to be greater than 0.0001 in. That is, in a measurement involving change of load, temperature and moisture, the corrected values of change of length are not likely to be in error by more than 0.0001 in. This value was determined by repeated checks of a number of cartridges under the conditions of calibration. In concrete the conditions of mounting are constant, so the accuracy of repetition at least should be greater than in calibration where the cartridge is removed from the test apparatus between readings. The sensitivity is an order higher than the accuracy, that is, about 0.00001 in. is the smallest change that can be detected readily with a portable bridge.

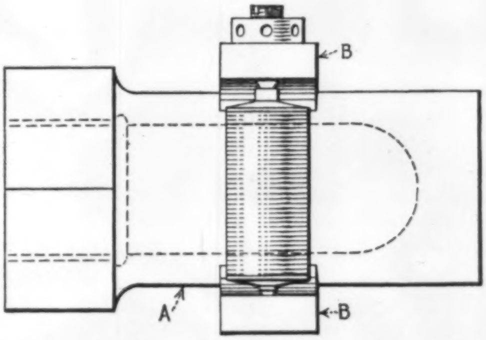

FIG. 11.—Diagram of Pressure Gage for High-Fluid Pressures.

Fluid Pressure Gage:

The general plan of a pressure gage for high-fluid pressures is shown in Fig. 11. It consists of a thick tube, *A*, which has two arms *B* integral with it, and long enough to permit mounting the carbon resistor between the ends at the side of the tube. When pressure is applied internally the tube expands, which causes the pressure on the carbon stacks to be released as the arms move apart. The calibration curve is practically the same as for the cartridge. One specimen designed for 0 to 20,000 lb. per sq. in. showed on test a change of resistance from 530 to 668 ohms, or 138 ohms total. Under average conditions of use the errors are not likely to exceed 3 per cent of the full range. As in the case of the cartridge a correction must be made if there is a change of temperature in the course of a measurement.

STANDARD FIRE TESTS FOR COMBUSTIBLE BUILDING MATERIALS

By M. E. Dunlap[1] and Frank P. Cartwright[2]

Synopsis

Standard fire tests of treated combustible building materials are desirable to disclose their relative effectiveness; to afford a control for their use in construction; and to secure uniformity of treatment. Such tests are not now generally in use, and are only partial in scope where used, but afford a basis for development. Standard tests should be related as nearly as possible to use conditions and requirements.

The assignment of a committee of the Society to develop such standard tests is recommended.

Need of Standard Test Procedure

For several reasons standard methods of testing the fire resistance of lumber and other combustible building materials are increasingly needed.

First, there are not less than 12 distinct processes or methods for rendering such materials fire-retardant, now in commercial operation or in process of promotion. These are of unknown but doubtless of varying effectiveness; both as to the extent, uniformity and permanence of fire resistance conferred, and their effects on strength, workability, and other pertinent characteristics.

It is desirable that those wishing to utilize treated materials, as for example, shingles, siding, flooring or plaster base, should be able to compare different products intelligently, with reference to their probable effectiveness in use; and that extravagant sales claims may be discounted.

Secondly, the selection of building materials assemblies on a performance basis for various fire-retardant functions is increasing. Combustible materials will enter into such assemblies to the extent that they are successful under test and economically feasible. Criteria are desirable by which the effectiveness of materials offered for field construction can be related to that of specimens tested in laboratory assemblies.

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[1] Engineer in Forest Products, U. S. Forest Products Laboratory, Madison, Wis.

[2] Engineer, National Lumber Manufacturers Association, Washington, D. C.

Thirdly, assuming that materials of a combustible nature, treated to confer varying degrees of fire resistance, will continue to be sold, some practical means of checking the uniformity and dependability of treatment will be desirable.

PRESENT TESTING PRACTICE

Only one set of tests is known to be in use as a practical basis for acceptance or rejection of fire-retardant lumber, namely, the set specified by the Bureau of Buildings of the Borough of Manhattan. No formal schedule of tests applying to other combustible construction materials is available. The Borough of Manhattan tests record the evolution of gas from a layer of shavings exposed to flame and the time needed for complete combustion; the percentage of cross-section of small samples burned away in a given period; and the persistence of combustion in a crib of small pieces after the heat source causing ignition is removed. They throw some light on rates of combustion, but fail to cover other points, such as the effect of fire temperatures on structural strength of the treated lumber, the rate of flame spread, the extent to which the material contributes to fire temperatures, the temperatures needed to cause and sustain ignition, and the moisture content of the lumber. These tests, furthermore, provide only for one type of use and do not recognize the possibilities of surface treatment.

Manufacturers have considerable trouble in meeting the requirements of the shavings test. The thickness of shavings or their character is not controlled, nor is their moisture content. The wood is brought to the laboratory where it may be planed off and used immediately or the shavings may stand a week awaiting test.[1]

Testing methods have been studied at the Forest Products Laboratory[2] relative to the inflammability of untreated wood and wood treated with fire-retarding compounds. One method consisted in exposing a small specimen 1 by 1 by 4 in. to a definite temperature in a hot tube and noting the time necessary for ignition and the loss in weight on burning. The other test was designed particularly for painted surfaces and shingles. The apparatus was equipped with a flat electrically-heated plate which subjected the lower end of the test panel to a definite temperature. The time required for ignition, area burned and period of flaming were taken as an indication of the inflammability of the wood.

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[1] Since the presentation of this paper the Borough of Manhattan test requirements have been revised and much improved.—ED.

[2] A detailed description of these tests will be found in a report, "The Inflammability of Untreated Wood and Wood Treated with Fire Retarding Compounds" by R. E. Prince, published by the National Fire Protection Association, 87 Milk Street, Boston, Mass.

These tests and those used by the Borough of Manhattan or some modification of them might be used as a basis for the development of a standard laboratory test.

The U. S. Bureau of Standards, Washington, D. C., in investigating combustible materials for interior finish and floors, is accustomed to apply rough tests for ignition, rapidity of combustion and flame penetration. The first is made by applying a standardized Bunsen burner flame to edges or corners and the second by igniting a specimen and exposing it to a regulated air current at ordinary temperatures or at 300° F. Observations are made of the readiness with which the samples burn, the time required for full or partial consumption, the time of flaming, the time of glowing, and the weight and character of residue.

In the Bureau's flame-penetration tests the central portion of a specimen is subjected to a standardized Bunsen burner exposure from below and observations are made of the temperature rise on the unexposed surface, the appearance of smoke and flame, and the lateral flame-spread from the burned-out area.

The Bureau of Standards also has developed tests for shingle decks which indicate their inflammability under various exposures. The equipment is quite large and is capable of furnishing any desired wind velocity or conflagration temperature. The tests are made by placing a small burning brand on the deck to start with and gradually using larger sized brands until the deck ignites. Observations are made on the passage of fire through the roof and on the rate of flame spread. This test is also used by the Underwriters' Laboratories at Chicago. It is too cumbersome for routine inspection work, although apparently quite satisfactory for studies of the inflammability of roof covering.

Manufacturers of fireproofing materials frequently make burning tests on small buildings to demonstrate the value of their treatments or paints under such conditions as the promoter sees fit to use. No accurate comparisons can be obtained by this method of test.

CHARACTER OF TESTS NEEDED

Combustible materials in construction are susceptible to fire damage in a number of ways:

1. They ignite when exposed to high temperatures of adjacent fires or from brands carried by the wind or hot air currents.

2. Fire penetrates through floors, walls and roofs, gaining access to the interior; or to other portions of the building.

3. Fire weakens lumber in the form of beams and columns, causing floors to drop; displacing plaster, and rendering walls unsteady.

4. In some instances fire is reported to spread along the surfaces of combustible materials in buildings rather than by convection of heated gases.

5. Burning materials add to the amount of heat generated in a building and therefore to the difficulties of fire extinguishment.

Obviously, comparative tests of fire-retardant treatments should indicate to what extent they will avoid these disadvantages. It is suggested, therefore, that a standard schedule of fire tests should include the following procedure:

1. Surfaces or decks of the material to be tested, preferably of commercial thickness, should be exposed to a fixed temperature, characteristic of conflagration exposure, and the periods necessary for ignition, and for combustion to penetrate the deck, should be registered in some suitable fashion.

2. Small beams and columns under rated live load should be exposed to temperatures characteristic of fires, and the period necessary for failure to occur, together with the proportion of section destroyed, should be noted.

3. The rate of flame spread along surfaces, under high and ordinary temperatures, should be registered.

4. A certain amount of combustible material, having a predetermined B.t.u. content, should be mixed with small fragments of the material to be tested and burned under conditions which allow determination of the heat volume contributed by the material.

There are two general types of fire-retardant treatments: surface and penetration. Both will probably find their place in the scheme of construction; the former being adapted mostly to materials which are delivered in fabricated form or in the sections in which they are to be used; the latter for materials subject to fabrication during erection. Their relative use in building operations also will be determined by the effectiveness of the treatment. Test procedure will probably have to be varied somewhat to take account of the different uses for which combustible materials may be adapted.

It would be desirable to work out tests to show the distribution of fireproofing salts in wood, similar to those used for zinc chloride and sodium fluoride by wood preservers. Wood which has been treated and split apart can be sprayed with certain solutions which change color upon coming in contact with the salt, indicating the areas treated and untreated.

Procedure preliminary to testing must allow for certain influential factors:

1. The samples should, of course, be selected by the investigator from commercial stocks.

2. Those treated by the penetration method should be not less than 3 to 5 ft. in length; the ends of lumber specimens should be removed preliminary to tests; and tests should be made both on finished and re-finished specimens. The species and the relation of heartwood to sapwood will be pertinent in the case of lumber.

3. Surface treatments to be applied on the job should be applied to test specimens by commercial painters or other craftsmen under the specifications recommended by the manufacturer.

4. Provision should be made for conditioning materials to uniform moisture content prior to testing.

5. There will need to be tests of the effect of treatment processes and materials on the strength, durability, moisture absorption, ease of working, painting properties, and other inherent characteristics of the material; and on the effects of chemicals on hardware and nails. These need not necessarily be included with a schedule of fire resistance tests, but can be made independently if desired.

For material to be treated and exposed to the weather there is needed an accelerated test which will disclose to what extent the treatment retains its efficiency under such exposure.

The current practice of testing fire-retardant materials by exposing them to the flame of a Bunsen burner or acetylene or gasoline torch is believed to have little significance, in that such intense forms of heat affect combustible materials quite differently from the temperatures met with in ordinary fires. It is not unlikely that the blow torch melts its way through the substance rather than burns it. In any case, it is doubtful whether the treatment chemicals react to such high temperatures in the same manner as when exposed to ordinary fire temperatures.

SUMMARY

Standard tests for fire-retardant lumber and other combustible materials are needed to make sure that the increasing demand for treated materials is properly supplied. Present testing methods afford a basis for development of standard tests but are inadequate for all purposes.

It is recommended that the American Society for Testing Materials assign a suitable group or committee to consider the requirements of the situation and develop standard test methods.

DISCUSSION

Mr. Markwardt.

MR. L. J. MARKWARDT.[1]—I think Mr. Cartwright and Mr. Dunlap have given us a very excellent paper in summing up the various methods that are used to make so-called fire tests on wood. To anyone who is familiar with wood, knowing its variations in properties and in moisture content, it is very easy to see how one might manipulate a test to show almost any preconceived conclusions, so one of the particular advantages, as I see it, of establishing standard tests would be to eliminate these variables that would otherwise get in and make the results subject to error and misinterpretation.

Again, wood frequently is regarded as a very flammable material, and we often fail to recognize that while it is flammable it transmits heat very slowly and offers a means of developing slow-burning construction.

I think Mr. Cartwright and Mr. Dunlap have made a very excellent suggestion when they propose that the Society enter this field.

Mr. von Schrenk.

MR. HERMANN VON SCHRENK.[2]—I wish to second the comments that Mr. Markwardt has made. I think this is a very timely presentation on a very important subject. As the authors have pointed out, our largest municipality has entered the field by requiring fire-retardant wood in many phases of construction, and in the absence of any very definite recommendation it has developed its own method for testing the degree of fire retardation. Those methods are good so far as they go, but, as the authors have pointed out, they are more of a beginning than anything else.

The subject is not an easy one. I remember some years ago I had occasion to enlist the interest of the British and German Fire Prevention Committees, none of which had any very definite way of determining degrees of fire resistance.

The first really practical scheme that was developed in this country was one developed by the Forest Products Laboratory, to which the authors have already referred.

Mr. Ingberg.

MR. S. H. INGBERG.[3]—This paper deals with a subject that in the past has not been given the attention its importance deserves. We have the two main divisions of construction into fire-resistive and

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[1] Assistant, In Charge of Section of Timber Mechanics, U. S. Forest Products Laboratory, Madison, Wis.

[2] Consulting Timber Engineer, St. Louis, Mo.

[3] Senior Engineer, U. S. Bureau of Standards.

Mr. Ingberg. non-fire-resistive, and the major effort of past work on fire-resistive properties has been given to materials and methods of application used for the former and newer building type.

That this was inevitable is apparent from the fact that the development of the fire-resistive building type lies chiefly within the past fifty years, and while improvements have been many, there yet remains much to be done before the application of the newer construction can be made on a sure quantitative basis to all general conditions.

Not considering building types consisting of combinations of fire-resistive and non-fire-resistive construction, improvements in making frame construction itself more fire-safe have been slow in finding application. Fire-stopping of open spaces within the construction and fire-resistive interior and exterior finishes are beginning to find recognition as effective means of retarding the spread of fire.

Many promising improvements have fallen by the wayside because no definite test or measuring stick was available for indicating their merits and which might form a basis for justifying any difference in cost as compared with the regular construction. Also investments have been made in materials and methods of application that probably give little added protection. The tests proposed, whose purposes are carefully outlined in the paper, are intended to supply such a standard of measurement. Designed for application directly to the material, they cover a field where fully satisfactory methods of measuring the fire hazard or fire-retardant effect have not been developed.

All fires start from small beginnings and the readiness with which they originate and attain initial spread can be much restricted by minor fire-retardant properties of adjacent materials, although these may be ineffective after the fires are beyond the initial stage. Some treatments of wood involving impregnation with chemicals apparently impart fire-retardant properties effective to varying extents after fires are beyond such initial stage. Properly designed tests should indicate the relative merit with respect to the untreated material and also give information on probable performance under the intended use in the structure.

Mr. Small. MR. A. R. SMALL.[1]—It occurs to me that the problem does not relate altogether only to wood. There is the related problem of fire-proofing, so-called, in textiles, hangings, etc.—accepted methods for determining the effectiveness of treatments of textiles that would be applied probably in a general way exactly as are the methods for determining effectiveness of treating wood. I heartily endorse Mr. Cartwright's recommendation that this subject be referred to the

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[1] Vice-President, Underwriters' Laboratories, Inc., New York City.

Mr. Small. Executive Committee of the Society for attention before determining that the project be entered upon. Its ramifications are many, as I see it. Opportunities for conflict are quite as many.

Everybody connected with the enforcement of fire prevention regulations knows, as the paper says, that in various communities and at various times in various ways there have been attempts to render wood finish less of a hazard to premises. Mr. Cartwright has presented the problem from the opposite point of view, arriving at the center of the circle just as quickly. Some of my contacts think this problem is one of making combustible building materials less hazardous rather than trying to make them fire retardants. We think of a fire retardant as a building method intended to confine fire and prevent it entering premises.

No doubt Messrs. Cartwright and Dunlap had in mind rather to make such uses of wood less objectionable than some have regarded it. It is a problem and will be a live one until some organization such as this Society works on it and produces the answer. I admire Mr. Cartwright for proposing the start.

Mr. Pierce. MR. DANA PIERCE.[1]—I have no disposition to do other than approve the enterprise and courage of an endeavor of this sort by the Society. I am, however, reminded of some of the problems that immediately come up for discussion whenever such matters are treated. To illustrate this, I note one or two phrases used in this report, such as "characteristic of conflagration exposure," "temperatures characteristic of fires," "high and ordinary temperatures," "temperatures met with in ordinary fires."

Those terms in the present state of the art cannot be defined with exactness for either the testing engineer or the fireproofer of wood. We do not know with exactness what are the temperatures characteristic of conflagration exposures nor do we know what are high and ordinary temperatures as applied to building fires.

It is in such difficulties as this that the chief obstacles lie in developing the test methods on a scientific basis. We have our standard method of fire testing for materials; whether it is suited to the testing of combustible materials as well as non-combustible is certainly open to question. The method is somewhat arbitrary in that we apply a standard method of fire, standard rate of rise of temperature, to the test specimen and observe the results without wasting time in discussing whether the temperatures and times involved are those that would exist in Louisville, Ky., or Chicago or Boston in an actual fire. Our art-of-fire-prevention study has not reached that point.

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[1] Underwriters' Laboratories, Inc., Chicago, Ill.

Mr. Pierce. These facts do not necessarily constitute an objection to the project we are considering, but simply indicate what seem to be fundamental difficulties which must be recognized. They are certainly practical questions. By people interested in a financial way in the rating of buildings, physical and chemical tests in the laboratory by whatever methods are naturally discounted somewhat as are also to a lesser extent tests of larger specimens—panels, walls, floors and so forth—such as have been conducted to a limited degree at the Bureau of Standards and Underwriters' Laboratories. These are real difficulties from the point of view of the practical man who wants something beyond a salesman's arguments. What the real results will be when the fire comes to a building containing fire-proofed wood still remains to be found out.

Three other points of importance should be noted. It seems to me that this excellent outline of the problem hardly gives enough emphasis to the question of permanency of fireproofing processes. The condition of the fireproofed materials and their fire-resistive ability months and years after their application and erection in buildings are certainly not the least important of the questions involved.

Secondly, the uniformity in securing the results aimed at—assuming that we have first determined what these results should be—is a factor which is of great practical importance.

Finally, there are differing problems in applying fire-resistive treatment to materials to be used for the siding of buildings, to shingles, to floors and general trim, not to mention others. The method of test, the spread of fire, the way fire reaches the material and the consequences of the fire will vary with their different application.

Those are all points which demand study both in the development of a test method, in the application of such a method and in the judgment of one material compared with another. They will have to be dealt with by whatever association or committee undertakes this important piece of work.

Mr. Emley. MR. W. E. EMLEY.[1]—It is not so many years ago when the test methods for the fire resistance of fire-resisting materials were perhaps in about the same chaotic condition, and, as I remember it, this Society has done quite a bit to bring them into the present generally accepted form to which Mr. Pierce has just alluded.

I believe we have a standing committee of this Society, Committee C-5 on Fire Tests of Materials and Construction, which has been to a large extent responsible for the selection, adoption, crystallization

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[1] Chief, Division of Fibrous and Organic Materials, U. S. Bureau of Standards, Washington, D. C.

I had better say, of a standard method for measuring the fire resistance of fire-resisting materials. As I listened to Mr. Cartwright's paper it struck me that by no very great stretch of the imagination one could easily imagine it within the function of Committee C-5 to develop new test methods whereby the low range of their scale might be magnified so that we could differentiate between a material which is distinctly combustible and one which is not so combustible. **Mr. Emley.**

Mr. Pierson. MR. C. H. PIERSON[1].—The title of the paper "Standard Fire Tests for Combustible Building Materials" would lead one to believe that the methods discussed were to be applied to buildings as a whole. It seems apparent from the text that the tests really apply to small specimens. It is questionable if the results obtained will be applicable to completed assemblies. As has been pointed out by the previous speakers, tests of small specimens may not necessarily give results which may be applied to commercial construction.

The ultimate use of all tests of this type, large or small, is to reduce the fire losses in buildings in which combustible materials are used assembled with other materials which may be either combustible or non-combustible. The problem is to ascertain how the combustible materials will perform when assembled as they are used commercially. The problem seems to be one of evaluating the fire hazard of the combustible material.

The tests of small samples are of value, first, in aiding an investigator who is attempting to devise a method of treatment which may prove of value when larger specimens are tested under the conditions in which they are actually used; second, tests of small specimens might be used to control factory production after a proved method of treatment had been attained; and third, tests of small specimens may be of value for use in check testing by inspection authorities.

Tests of small specimens appear to be of interest for research work, for commercial control or independent inspections.

Mr. Ingberg. MR. INGBERG.—I want to second what has been said here relative to the necessity for proper interpretation of results. Using our regular fire testing procedure we are at present testing combustible or partly combustible constructions and obtaining certain results without any generally accepted interpretation as to what they mean when the constructions are applied in buildings.

Mr. Cartwright. MR. F. P. CARTWRIGHT.[2]—So much has been said and so well said that I have very little to add. I think we are indebted to the

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[1] Assistant Engineer, Protection Department, Underwriters' Laboratories, Inc., Chicago, Ill.

[2] Engineer, National Lumber Manufacturers' Association, Washington, D. C.

Mr. Cartwright. remarks which the various representatives of underwriting interests present have made. Their experience in these matters is valuable and their efforts are directed in a somewhat different line than mine, which naturally leads them to look with misgivings on such a proposal.

One of the questions raised concerned the temperature of an ordinary fire, a conflagration exposure, etc. I think we are in perfect agreement on that point, that there is no one temperature of which it can be said that it is representative of the temperatures which will obtain in a building enclosure when the fire occurs.

The study which has attended the development of the time-temperature curve, however, and the reasonably close check on that curve which has been afforded by the burn-out tests at the Bureau of Standards, leads me to think that if the proper talent is brought to bear, temperatures can be selected for these tests which will at least give us a dependable comparison between materials and treatments.

As to the uniformity of the materials involved, I do not think there is greater variation on the whole in lumber of a given species or grade than obtains in many of the materials which are used for fireproofing purposes, so-called, as, for example, concrete. The difference between the fireproofing qualities of concrete made with quartz aggregate and that made with limestone aggregate is a fair example, I think.

As to the permanence of the treatments, that of course is a necessary consideration, and I regret that I do not seem to have given the matter sufficient stress in this paper. For the most part I contemplate that such treatments would be employed on the material used on the interior of buildings and the weather effects in that case would be relatively slight.

I will but read the last paragraph of the paper: "It is recommended that the American Society for Testing Materials assign a suitable group or committee to consider the requirements of the situation and develop standard test methods."

I will move, Mr. Chairman, that this recommendation be referred to the Executive Committee for its consideration.

[The motion to refer to the Executive Committee the recommendation that the Society consider requirements for fire tests of combustible building materials was put to vote and carried.—ED.]

Mr. Ingberg. MR. INGBERG (*by letter*).—In the paper there are two references to our work that were passed over on the first reading that apparently should have been given attention.

As concerns the tests for combustible partition, ceiling and floor finishes, their use has been confined to comparisons of a material

Mr. Ingberg.

proposed for a given use with another, either specifically approved or disapproved for the same use by enactment or ruling. Samples of the materials 6 in. square of comparable thickness and conditioned at the same temperature to constant or nearly constant weight are tested with match and gas flames applied to the corners, edges and sides to determine susceptibility to ignition from small flames in contact for brief periods, and the extent to which burning continues after removal of the flame. This is not intended as a test to determine the temperature required for ignition, which depends on the form and size of the sample, the air supply and the duration of the heat application. We believe, however, that the tests give with a sufficient number of repetitions pertinent information on the relative extent to which such materials contribute to the origin of fire. The other tests give information on the contribution to the spread of fire along the surface and through the thickness as of ceiling or partition finish. The indicated temperatures produced immediately under the sample by the Bunsen burner as applied by us in the flame penetration tests have obtained at points under the ceiling in our burning-out tests with office occupancy within five minutes after the start of the test. While in common with most tests with small specimens of materials entering into larger constructions, those outlined give mainly relative values, we have otherwise no apologies to make for the methods employed.

Relative to the term "conflagration temperature" as used in the paper, what the authors mean is, without doubt, the elevated temperature of the air currents applied in some of our tests. It has been observed in building and forest fires and proved in tests, that combustible materials will ignite from smaller brands or flames and burn and spread the fire more readily when the surrounding air is at temperatures considerably above normal. In description of large fires this is often called the "hot blast," although the same effects are probably present to some extent in all fires that are past the initial stage. While surrounding a burning area the temperatures vary from that in or immediately above the burning buildings or materials, to ordinary air temperatures some distance away, we have in our tests chosen 300° F. as representative of the average for a zone of temperatures that do not cause immediate ignition but facilitate combustion. The size and shape of the area over which they obtain depend on the size and intensity of the fire, the kind of buildings or materials burning and the prevailing wind. In our tests of combustible roofing materials we have found brands applied with air currents at 300° F. to be as destructive as those many times as large applied with air currents at ordinary temperatures.

Mr. Ingberg. "Conflagration temperature" as used in the paper apparently does not mean the fire temperature, concerning the degree of which for either conflagrations or other fires we have made no commitments, and which should not be confused with what we obtain from our burning-out tests, the latter being expressed in time-endurance units as referred to the recognized time-temperature control curve for fire tests. To avoid any confusion we generally designate the tests concerned as being made "with air temperatures at or near 300° F."

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